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The Physical Nature of the Shrinkage of Portland Cement.

By H. B. SEED, B.Sc. (Eng.)

THE phenomenon of shrinkage is of considerable importance in the design and æsthetic qualities of concrete structures, and a great deal of experimental and theoretical work has been carried out with the object of estimating the magnitude of the movement and its effects. These investigations show that in most structures the effects in design are negligible, but that in long continuous concrete spans some allowance has to be made. This generally takes the form of subdividing the length into sections in order that cracking due to shrinkage shall be prevented, or at least reduced.

Cracking caused by failure of concrete in tension due to differential shrinkage, or to shrinkage under partial or complete restraint, is the one effect of shrinkage of real concern to the engineer. The problem is still receiving attention, but it is likely that no marked improvements can be expected until engineers are provided with a complete and simple picture of the mechanism of the shrinkage movement. It is with this object that the present paper has been prepared.

In order to appreciate the nature of shrinkage some knowledge is required of the composition of concrete, and it is thought desirable to summarise the description by Mr. C. G. Lynam[1] of the changes occurring during the setting and hardening periods. On this subject there are two basic theories differing on which of the chemical substances formed after the addition of the mixing water is mainly responsible for the properties of the concrete. According to the crystal theory the final products are silica gel and calcium hydroxide, of which the latter is the more important. Thus, due to the contraction which takes place when calcium hydroxide is formed by hydrolysis, the particles of aggregate are held in position by a surrounding mass of interwoven crystals. The gel theory argues that the proportion of water generally added would dissolve very little lime,

and that the hydrolysis occurring is therefore small ; crystals of tricalcium silicate are formed, but the cement is a hardened gel of calcium silicate.

A more general, and more likely, theory reconciles these two ideas by saying that on hydration a gel is formed which, after a considerable time, is replaced by crystals. As a rule the formation of a gel is preceded by the formation of a colloid solution, that is a suspension in a liquid of fine particles ranging from microscopic to molecular. Owing to their small size and high ratio of surface area to mass, the forces acting on the particles, for example, surface tension and electrical attraction and repulsion, can overcome the effects of gravity and the particles are held in suspension. Under certain conditions, such as change of temperature, loss of moisture by evaporation or, particularly, the addition of an electrolyte, the colloid coagulates or stiffens, forming a gel. In this comparatively rigid state the gel is still capable of holding a considerable quantity of liquid ; silica gel may hold up to 300 molecules of water to one of silica, though much of this may be lost by evaporation and up to 90 per cent. may be expelled under pressure. This loss of moisture causes the gel to harden, but the change is by no means irreversible. Owing to the continuous diffusion of liquid through a gel, liquid may be added or removed as external conditions dictate, causing the gel to swell or shrink. Under normal conditions, however, most gels lose moisture by evaporation, hardening is promoted, and the gel assumes a crystalline form.

When water is added it begins to dissolve the surface of the cement particles, forming (*a*) a colloid and (*b*) a series of solutions. As solution progresses heat is evolved, causing the colloid to form a gel, that is the concrete starts to set. For this reason hot water hastens the setting of cement. Meanwhile more water attacks the cement particles, and the mixture consists of colloid, gel, solutions and free water which has not yet reached the surfaces of the particles. This water may be squeezed out of the concrete while casting or lost by evaporation during setting. New colloid, as it is formed, is adsorbed by the gel, but when the mixture begins to dry out the solutions evaporate and crystals of calcium sulpho-aluminate and calcium hydroxide are formed in the minute crevices or micropores which are present in the gel structure. As the water is used the gel hardens, but since the drying process continues for a long time a certain amount of free water is invariably available.

Thus the water content of concrete can be sub-divided into three main types : (*a*) Firmly held or chemically combined water as in $Ca(OH)_2$, (*b*) Loosely held such as colloidal water or water of crystallisation ($CaSO_4 . 2H_2O$), and (*c*) Mechanically held or free water. The proportion of free water, initially high, is reduced considerably by losses in evaporation, the constant hydration of cement, the conversion of colloid to gel, and the formation of crystals. As a result the water becomes more firmly held with age and the gradual crystallisation causes a corresponding slight change in the properties of the concrete, an increase in strength, and a reduction in elastic strain under stress.

The gel constituent, after drying to a certain stage, begins to develop a capillary structure giving easier movement to the water, which, attracted to the surface,

dissolves some calcium hydroxide and deposits it in the capillaries and at the surface. Near the surface carbon dioxide from the air converts the hydroxide to carbonate, and if this blocks the channels no further diffusion can occur. If it does not, however, more water and carbon dioxide dissolve the carbonate, lime is carried to the surface, and the passages are left free as before. Macropores or small cavities, due mainly to inadequate mixing and packing or to lack of cement, are connected by the capillaries, giving a large surface exposed to this solvent action with the result that calcium hydroxide may be gradually lost and the cement gels weaken. Such an occurrence is rare since moist curing fosters the production of colloid and helps unhydrated cement to combine with available water, forming more gel and crystals and so making the structure more stable and dense and the capillaries smaller. Further, it discourages the flow of water from the concrete causing calcium hydroxide or carbonate to block the channels; the movement of the water then diminishes rapidly, the growth of crystals and gel diminishes it further, hardening is promoted, and the strength increases. But with initially large pores and atmospheric conditions promoting the flow of water the concrete may deteriorate.

With a high water-cement ratio, excess water dissolves the lime and leaves a gel of silica instead of calcium hydrosilicate, making the concrete weaker. A low water-cement ratio promotes a rich colloid before gel formation, giving a dense structure afterwards, a reserve of unhydrated cement to combine with any free water, and increased strength.

The hardening process is slow and continuous. It is suggested that the initial set corresponds with the formation of the gel and the final set marks the time when the gel has hardened sufficiently to bear an arbitrarily fixed load. Good concrete, however, increases in density and strength for many years through the hydration of its reserves of cement.

Shrinkage.

As we have already seen, the addition of water to Portland cement results in the formation of a gel and the property of gels when wetted to take up liquid and swell, or when dried to give up liquid and contract, has been firmly established. If, then, water is removed from the gel constituent of a concrete, a contraction of volume is to be expected.

When water is added to Portland cement there appear to be three phases of volume change, namely, (*a*) initial expansion, (*b*) rapid contraction over a short period, and (*c*) a slower and progressive contraction over a number of years.

The second movement, a relatively rapid contraction which takes place mainly over the setting period, is due to volume changes caused by the chemical combination of the water with the cement constituents. It is not connected with loss of moisture—this has been clearly demonstrated by measuring the volume change while the mixture was immersed in benzene—and is known therefore as autogenous shrinkage. Although the hydration of more cement particles continues

over an indefinite period the effects after the initial hydration are so small as to be negligible, and subsequent expansion or contraction is almost entirely due to adsorbtion by, or loss of water from, the gel. Tests by Mr. J. C. Pearson[2] at the U.S. Bureau of Standards in 1921 showed moderate expansion or contraction over the first four hours while the mortars were still plastic, followed by a pronounced contraction during the initial set (autogenous shrinkage). The rate of shrinkage was reduced considerably at eight to ten hours during the time of final set, very little change occurred from twelve to twenty-four hours, but thereafter a long continuous shrinkage began. It is the last progressive volume change which is of importance to engineers.

Mortar retains the property of expanding and contracting on wetting and drying after many years, and it is clear that all movement in concrete must take place in the cement paste. Cement gel at the surface loses water by evaporation and replaces some of this from the moister concrete inside, so forming a moisture gradient across the concrete. Since the volume of the gel is a function of the moisture content, shrinkage will vary across the concrete, being greatest at the outer surface and least at the central axis. In consequence, internal stresses are set up—the skin tends to contract more than the core, and this movement puts the skin in tension and the core in compression.

Differential shrinkage has been demonstrated both quantitatively and qualitatively by numerous investigators. For example, Dr. A. D. Ross[3], using a rectangular prism coated with pitch on all except one of its faces to represent a 12 in. slab of great dimensions in plan, found that the difference of linear shrinkages at the surface and at the centre amounted to 470×10^{-6} inch at 200 days. With $E = 3 \times 10^6$ lb. per square inch, this represents a stress difference of 1,400 lb. per square inch, and while creep would undoubtedly operate to reduce this figure it is clear that surface cracking due to failure of the concrete in tension will result in unfavourable cases.

When concrete is wetted these conditions are reversed, the core is in tension and the skin in compression though the specimen lengthens as a whole. Such changes may be reversed many times without any permanent effect on the linear dimensions. Tests by Mr. A. H. White[4] and others have shown that up to ages of at least twenty years mortars and concrete respond quickly to variations in moisture conditions, although the rate of change may not be so great as in a freshly-cast specimen. Volume seems to depend less on the quantity of water than on the manner in which it is held, free water having practically no effect on the linear dimensions. It has been found that concrete takes up a certain amount of water before it begins to expand and goes on expanding slowly after it has ceased to absorb water. Mr. A. H. White[5] also found that concrete will take up a fair weight of benzene without any effect on the linear dimensions, and since this could not be chemically combined or adsorbed by the colloid it could only have been stored in the capillary channels or micropores of the concrete. This implies that free water has no effect on movement, which is mainly due to

change of moisture content of the gel or to its subsequent conversion to crystals. It suggests, too, that a wet concrete will lose a certain amount of water before it begins to contract. That this is the case was shown by Dr. Ross, who reported: "In all cases a marked reduction in the rate of loss of moisture was noted after a short period while the decrease in the rate of shrinkage was more gradual and continuous. This is in accordance with the conception of an initial abundance of free water which is easily lost by evaporation at the surface giving initially a steep gradient to the moisture loss curve." It has been suggested by Mr. H. W. Coultas[6] that, in the later stages of drying, shrinkage and moisture loss are proportional, but this was not supported by Dr. Ross's results. In this connection he says, "Although there was some indication in certain cases, no general proportionality was evident. Since moisture distribution is three dimensional it seems improbable (except in cases of unique size and shape) that there could be such a proportionality, although it is not impossible that such a relationship might be found for volumetric shrinkage."

Although alternate wetting and drying may cause no permanent change in the dimensions of concrete it is not without effect on the physical nature of the material. Mr. T. Matsumoto[7] found that a specimen that has contracted in size may have increased in weight. During the wetting periods some of the water is used to hydrate more cement, the combined moisture content is increased with a consequent formation of more crystals, and on drying a greater proportion is retained as water of crystallisation or chemically combined in the gel. With the formation of more colloid the gel is gradually enriched, crystals grow and by their pressure harden the gel, and the circulation of water induced in this way leads to more complete hydration of the cement.

Since concrete expands on wetting, and the contraction on drying may induce stresses sufficiently high to cause minute failures (evidenced by crazing), it would seem clear that an attempt could be made to reduce the shrinkage by storing the concrete during its early life in a moist atmosphere. Moist curing, by flattening the moisture gradient, reduces differential shrinkage and at the same time enriches the surface gel. Thus internal stresses are kept low while the gel grows strong enough to resist them, leading to a more uniform and stronger concrete.

There has been a great diversity of results in shrinkage experiments, due no doubt to lack of appreciation of the number of factors involved, but a considerable amount of work has been carried out on the subject and the following is based on the reports of Mr. R. E. Davis[8] and Dr. W. H. Glanville[9].

(*a*) TYPE OF CEMENT.—Volume changes vary considerably with chemical composition and are also affected by the fineness of grinding. Dr. Glanville showed that shrinkage was greater for high-alumina cement than for rapid-hardening Portland cement, while the latter in turn contracted more than normal Portland cement. There were differences, too, in expansion due to wetting, for while Portland cement concretes showed continuous swelling in water, high-alumina cement concrete showed a slight tendency to shrink at first followed by a gradual swelling. This seems to confirm tests by Mr. W. K. Hatt,[10] from which he concluded that

cements which exhibited the greater net expansions under water also developed less net contractions in air.

Mr. P. H. Bates [11] found greater expansion in water with fine cements, the expansion of normal cements being about 90 per cent. that of reground cements. This can be accounted for by the greater surface area available for hydration in the fine cements, with consequent formation of more gel. On the other hand, accelerated expansion tests reported in *Le Ciment* (1923), using the Le Chatelier apparatus (the cement paste being boiled for six hours), showed decreased expansion with increase in fineness. Other results seem to support Mr. Bates's results, but on the whole the effects of fineness of cement appear to be very small.

(*b*) PROPORTIONS OF MIX.—Mr. F. R. McMillan[12] and Mr. Matsumoto[7] both showed that the richer the mix the greater the shrinkage, the latter also showing this to be true for expansion. These results have since been confirmed by Messrs. Davis and Troxell in California, and again the most probable explanation is the increase in potential shrinkage caused by an increase in the gel content. An interesting feature of Mr. Matsumoto's results was that the contractions after 58 days in air did not bear any consistent relationship to the cement content, while ultimate shrinkages showed a clear relationship. This suggests that the results of short-time tests are not always a criterion of the ultimate values.

O. Graf[13] pointed out that increase in cement content is not always accompanied by increase in shrinkage because a greater quantity of water is required for the lean mix in proportion to the cement, and the resulting increase in shrinkage may outbalance the decrease in shrinkage caused by the use of less cement. This may be the more usual case, but it introduces a second variable, the water content, which itself may be masked by the influence of some other factor. In view of this, Graf's conclusions are not contradictory to other results.

(*c*) TYPE AND GRADING OF AGGREGATE.—The influence of the type of aggregate was shown by Mr. R. E. Davis whose results demonstrated the small contraction of granite and limestone concretes in comparison with similar gravel and sandstone concretes, but the effect of variation of grading is not nearly so marked. Despite the fact that mortar mixes of a given water-cement ratio are subject to greater volume changes than concrete mixes of the same water-cement ratio, Messrs. Davis and Troxell[14] concluded that the percentage of fines did not appear to affect the ultimate volume changes. Investigations by Mr. Hatt and Dr. Glanville, however, indicate that while the effect is negligible the use of aggregates with higher surface moduli will, if anything, result in slightly greater volume changes.

(*d*) ADMIXTURES.—Until recently the only available information on the effects of admixtures was that obtained by Messrs. Davis and Troxell [14] showing that (*a*) the addition of lime has very little effect on the shrinkage movement and (*b*) the results are affected by replacing cement by, say, 10 per cent. lime or clay. For a lime replacement shrinkage is reduced, while for clay it is increased.

Although it is impossible to exclude the possibility of direct effects of these materials, it is probable that the differences in shrinkage values resulting from such substitutions are of a secondary nature. Thus, a lime replacement gives a leaner mix, a proportion of the mixing water is required for hydration of the lime so reducing the effective water-cement ratio, and the lime itself hinders the access of water to the cement grains—all contributory causes to a reduction in gel formation and therefore in shrinkage. A clay replacement will act simply as an aggregate which may take up a slight quantity of water, but which will then itself become subject to shrinkage. Thus the effect on the mix as a whole is a reduction in cement content, no appreciable change in water content, and therefore an increased water-cement ratio. If the increase in shrinkage due to this higher proportion of water in the gel exceeds the decrease resulting from the reduction in gel formation, the mix will exhibit the higher volume changes found by Mr. Davis—a result depending on the proportion of cement replaced.

Further information has recently been published by Mons. Lossier[15] who has found it to be possible to produce cements with controlled expansions by adding to normal cement (*a*) a sulphoaluminate cement which acts as the expanding agent and (*b*) a stabilising element, usually slag cement, which is capable of stopping the expansion but whose action is retarded. By using these materials it is possible to induce in the cement an artificial expansion and, further, to regulate the expansion in magnitude and in duration. The mechanism of the movement is not understood, but its magnitude for cement paste may be as much as 5 per cent. developed over a minimum period of 24 hours or a maximum of about 30 days. Once the expansion has ceased, normal shrinkage properties are exhibited and the expansion is gradually reduced. Expansive cements of this type are manufactured in two principal qualities. First, a cement with comparatively small expansion in which the maximum initial expansion is regulated to about 0·003 in. per inch. This value is approximately equal to the free shrinkage, so that on drying the initial length is gradually restored. In the second quality the initial expansion is four or five times this amount, and an increase in volume is therefore permanently retained. The development of these cements is a step forward in the control of shrinkage movements. Unfortunately, however, such improvements are rarely effected without introducing undesirable features. Thus, expanding cements are found to be more subject to deterioration than Portland cements, the sand and aggregate with which they are mixed must be free from sulphates, and they are not, as yet, resistant to the action of sea-water.

(*e*) MOISTURE CONDITIONS.—Dr. Glanville, Mr. R. E. Davis and others found increased shrinkage with higher water contents, and that the action of concrete in a damp atmosphere depends on the dryness of the specimen. If the humidity of the atmosphere exceeds that of the specimen the concrete will expand, and if not the concrete will contract ; hence the use of moist curing to prevent contraction at early ages. Investigations seem to show that moist curing does, in fact, produce only a negligible reduction in shrinkage. Mr. McMillan's tests, for

example, showed that while the rate of shrinkage of a moist-cured specimen was reduced, the ultimate value was not affected; but a more comprehensive result was obtained by Mörsch[16] who measured the expansion due to moist curing as well as subsequent shrinkage. The final contraction of a specimen stored in air from the date of casting was found to be 0·032 in. A second specimen from the same mix was water cured for 196 days, when it showed an expansion of 0·032 in.; it was then allowed to shrink freely in air, the ultimate length showing a total expansion 0·004 in., that is a shrinkage of 0·028 in. Thus, although moist curing does not affect shrinkage appreciably, it does affect the actual length of the specimen. Its main influences are the prevention of shrinkage until the gel is sufficiently enriched to ensure freedom from minor disorganisations and the decrease in internal stress resulting from the reduction of differential shrinkage.

(*f*) AGE AT FIRST OBSERVATION.—When the final set has taken place, at say, the age of one day, the main proportion of water in concrete is that contained in the colloid or gel. As moisture is lost by evaporation the rate of moisture loss decreases. Since the volume of the gel is a function of its moisture content, the rate of shrinkage must decrease with time. Also water is lost from the gel by its conversion into chemically-combined water or by the formation of crystals. This reduces the evaporable moisture content and leads to further reduction in the rate of shrinkage. Thus, although the rate may be fairly steady over the first two weeks, there is from this time on a continuous reduction until, after one year, movement has almost ceased, and a graph of shrinkage against time is approximately exponential in form. Dr. Faber's results[17], for example, for the average shrinkage of concrete mixtures ranging between 5 : 1 and 6 : 1 showed the contraction after three weeks to be one-half and after ten weeks three-quarters of the contraction over the first year. The recorded shrinkage of concrete depends therefore on the age at which the first reading is taken—the older a specimen the lower will be its subsequent shrinkage.

(*g*) LENGTH OF PERIOD OF INVESTIGATION.—Provided moisture conditions remain constant or nearly so, the shrinkage of moist concrete in a dry atmosphere, or the expansion of dry concrete in moist atmosphere, is progressive and continuous though at a decreasing rate. The shrinkage therefore increases continuously with time. Mr. White showed this continuous expansion and contraction over a period of ten years, though changes after one year were very small compared with those previously recorded.

(*h*) SURFACE-VOLUME RATIO.—Shrinkage is considerably affected by the size and shape of the specimen on which it is measured, and a comprehensive investigation of this effect has been published by Dr. A. D. Ross[3] from which the following conclusions are taken. Previous results indicated that the smaller the specimen the greater the shrinkage, and tests on specimens of varying sizes confirmed this. It was, however, from tests in which the shape was also varied that the main results was obtained. These showed that shape is a less important factor than surface-volume ratio; that for specimens of the same mix drying under the same conditions the magnitude of the shrinkage is a function of this ratio; and that

the variation may be very considerable—possibly several hundreds per cent. in the case of a large structure on the one hand and a small test specimen on the other. Dr. Ross's results show that for surface-volume ratios of 1·2, 2·2, and 3·1 the maximum skin shrinkages for the concrete were 100×10^{-5}, 108×10^{-5}, and 120×10^{-5} respectively, that is the lower the ratio the lower the shrinkage. Since in general, and certainly for specimens of the same shape, the larger the specimen the lower its surface-volume ratio the earlier conception that large specimens exhibit lower shrinkages is not surprising. The explanation is the changing nature of the moisture content. In a specimen with a large surface-volume ratio evaporation of the free water and the moisture in the gel is fairly rapid, the equilibrium position is reached at an early age, very little water is available for the further hydration of cement, and only a relatively small proportion can attain the irreversible chemically combined or crystallised form. In the interior of a mass of small surface area, however, moisture loss is greatly impeded, the rate of shrinkage is reduced, and contraction continues over a longer period. This promotes increased hydration and a gradual conversion of gel into crystals, more water reaches the irreversible stage, and so the ultimate moisture loss and shrinkage are reduced.

(*i*) ABSORPTION OF MOULD.—Messrs. Pearson,[2] Davis and Troxell[14] showed that shrinkage is less when mortars are cast in absorbent moulds. This is a secondary effect, the main cause being a reduction in the effective water-cement ratio resulting from the loss of moisture to the mould, with possibly a certain amount of unrecorded shrinkage before the moulds are removed. Earlier investigations showed a considerable variation amounting to several hundreds per cent. in the values of shrinkage and expansion—in fact a report by Mr. Hatt[10] showed a variation of 400 per cent. in his own results. The chief reason for this was a lack of appreciation of the variables affecting the results and the fact that no allowance was made for them. Marked differences were also due to differential shrinkage. Measurements in some cases were made on the skin and in other cases on the core without regard to the different rates of movement across the section. More recent tests have been made under controlled conditions with a considerable increase in consistency of the results. Even so it would appear that variations in comparable tests up to 40 per cent. are possible. Although these tests were conducted under laboratory conditions they provide some indication of the factors likely to be encountered in practice. With the development of expanding cements it is probable that tensile shrinkage stresses in reinforced concrete will ultimately be eliminated, but tensile stresses will continue to be set up in the concrete of unrestrained slabs due to differential shrinkage no matter what type of cement is used.

Thus if cracking is to be reduced as much as possible an attempt must be made to reduce the magnitude of shrinkage. This may be effected by (*a*) the use of normal Portland or Portland blastfurnace cement, (*b*) avoiding too rich a mix, (*c*) using granite and limestone aggregates, (*d*) keeping the water-cement ratio as

low as possible; this also leads to an increase in strength, and (*e*) an adequate period of moist curing. Of importance, too, in the development of shrinkage stresses is the relief of stress due to creep. To this end it is desirable to use concretes exhibiting high creep movements, but unfortunately this is not always compatible with a reduction in shrinkage.

[1] Growth and Movement in Portland Cement Concrete. C. G. Lynam. *Oxford University Press.*

[2] Shrinkage of Portland Cement Mortars. J. C. Pearson. *Journal American Concrete Institute*, 1921.

[3] Size, Shape, and Shrinkage. A. D. Ross. *Concrete and Constructional Engineering*, Vol. XXXIX, No. 8. August, 1944.

[4] Volume Changes of Portland Cement as Affected by Chemical Composition and Aging. A. H. White. *Proceedings American Soc. Testing Materials.* 1928.

[5] Hydrated Portland Cement as a Colloid. A. H. White. *Colloid Symposium, U.S.A.* Vol. 5.

[6] The Effect of Coarse Aggregate and other Factors on the Properties of Concrete. H. W. Coultas. *Structural Engineer*, March, 1936.

[7] Study of the Effects of Moisture Content on the Expansion and Contraction of Plain and Reinforced Concrete. T. Matsumoto. *Bulletin of the University of Illinois Expt. Station*, No. 126, 1921.

[8] Volumetric Changes in Concrete. R. E. Davis. *Journal American Concrete Institute*, 1930, pp. 407-443.

[9] Shrinkage Stresses. W. H. Glanville. *Building Research Station Technical Paper*, No. 11.

[10] Physical and Mechanical Properties of Portland Cements and Concretes. W. K. Hatt. *Purdue Univ. Eng. Expt. Station, Bulletin* 34, 1928.

[11] The Effect of Finer Grinding and a Higher SO_3 Content upon the Physical Properties of Portland Cement. P. H. Bates. *Proceedings American Society Testing Materials*, Vol. 15, 1915.

[12] Method of Designing a Rectangular Reinforced Concrete Flat Slab. A. C. Janni. *Trans. American Soc. C.E.*, Vol. 80, 1916. See discussion by F. R. McMillan.

[13] Shrinkage of Prisms of Cement Mortar of Different Proportions. O. Graf. *Zement*, 1929, 15(35).

[14] Volumetric Changes in Portland Cement Mortars and Concretes. R. E. Davis and G. E. Troxell. *Journal American Concrete Institute*, Vol. 25, 1929.

[15] Cements with Controlled Expansions and their Applications to Pre-stressed Concrete. H. Lossier. *Structural Engineer*, 1946.

[16] Der Eisenbetonbau. Mörsch; Konrad Wittwer, Stuttgart, Germany.

[17] Plastic Yield, Shrinkage, and other Problems of Concrete and their Effects on Design. O. Faber. *Proc. Inst. C.E.*, Vol. 225, 1927-28, Pt. I.

Acknowledgment.

Two articles in the May, 1946, number of this journal, entitled "New Cement Works in Sweden" and "Fuel Consumption of Lepol Kilns," were abstracts of articles which originally appeared in our United States contemporary "Rock Products." We regret that this acknowledgment was inadvertently omitted.

Manufacture of Cement and Sulphuric Acid from Anhydrite.

A GERMAN PROCESS.

A REPORT describing the manufacture of cement and sulphuric acid from anhydrite by I. G. Farbenfabrik at Wolfen, Saxony, has been prepared by Messrs. A. H. Loveless, R. J. Morley, and W. B. Perrycoste for the British Objectives Intelligence Sub-committee and published by H.M. Stationery Office (B.I.O.S. Report No. 678, Item No. 22. Price 3*s.* 6*d.*).

The authors state that so far as is known this process is worked at only three other places in the world. The first plant was erected at Leverkusen (Germany), the second at Billingham (England), and the third at Miramas (France). Two kilns and four contact systems were built at Wolfen in 1938 and two more kilns and four more contact systems were built in 1942. The Wolfen plant, like those at Leverkusen and Billingham, starts from anhydrite, very pure in the case of Wolfen, containing over 98 per cent. $CaSO_4$. The Miramas plant was said to use gypsum, $CaSO_4 . 2H_2O$.

The basis of the process is the reduction of calcium sulphate with coke:

$$CaSO_4 + \tfrac{1}{2}C = CaO + SO_2 + \tfrac{1}{2}CO_2 \text{ (about 1200 deg. C.)}.$$

To facilitate the reaction, enough clay is added to lead to the formation of Portland cement clinker in place of CaO, the reaction being represented approximately by

$$CaO + \tfrac{1}{8}Al_2O_3 . 2SiO_2 = \tfrac{1}{4}\,3CaO . SiO_2 + \tfrac{1}{8}\,2CaO . Al_2O_3 \text{ (about 1400 deg. C.)}.$$

The fundamental reaction is thus modified by using a higher ratio of $SiO_2 : Al_2O_3$ (about 2 parts by weight of SiO_2 to 1 part by weight of Al_2O_3) and by adding Fe_2O_3 as a flux. In this manner a Portland cement clinker is simultaneously produced in place of the valueless lime. SO_3 formation proceeds catalytically with vanadium catalyst according to the equation $2SO_2 + O_2 = 2SO_3$. Sulphuric acid is formed by addition of water to the sulphur trioxide: $SO_3 + H_2O = H_2SO_4$.

Raw Materials.

The process requires anhydrite, clay, coke, sand and pyrites cinders.

CLAY.—The raw clay is dried at about 120 to 150 deg. C. concurrently with producer gas. Of the total free water content of 28 to 30 per cent., about 20 per cent. present in the form of free water is driven off, while the remainder remains in the dry clay as combined water. The analysis of the dry clay is: H_2O (bound), about 10 per cent.; SiO_2, 49 to 53 per cent.; Al_2O_3, 30 to 35 per cent. (of which about 4 per cent. is TiO_2); Fe_2O_3, about 3 per cent.; CaO, about 1 per cent.

ANHYDRITE.—The anhydrite is crushed at the quarry to about 50 mm. size and further reduced at the works to 20 to 30 mm. If the anhydrite is delivered in a moist condition it is dried before reaching the crushing plant. The analysis of the anhydrite is: H_2O, 0·2 to 3 per cent.; $CaSO_4$, 96·5 to 99·5 per cent.; SiO_2, 0·1 to 0·2 per cent.; MgO, 0·1 to 0·2 per cent.

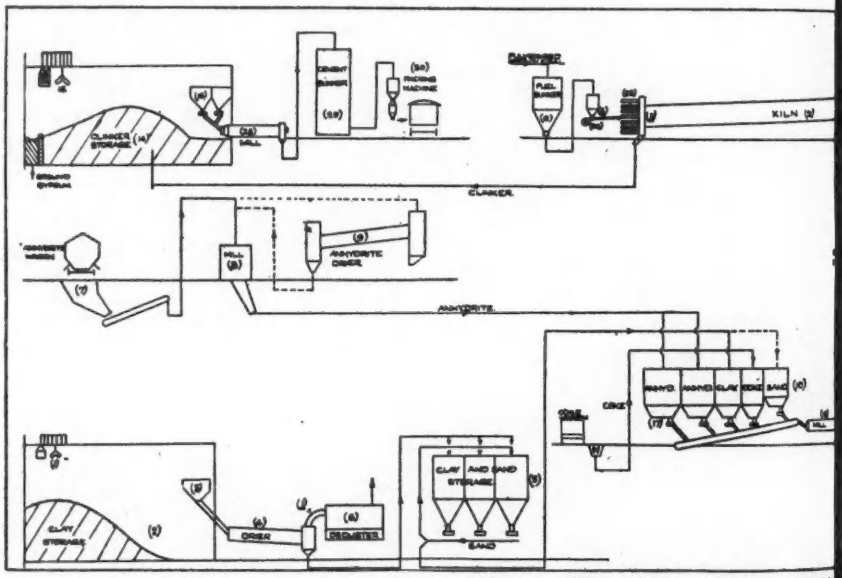
SAND.—The sand is dried in the clay drier to a moisture content of 5 per cent. The coarser sizes (from about 30 mm. upwards) are retained on grids at the drier

outlet. The analysis of the dry sand is: SiO_2, 89 to 94 per cent.; Al_2O_3, 4 to 6 per cent.; CaO, 0·5 to 2 per cent.

PYRITES CINDERS.—Since the raw materials do not contain enough iron, a small amount must be added in the form of pyrites cinders, which is obtained from the pyrites sulphuric acid plant at the works. The cinders are passed through the drier with the sand. During this operation the addition of the cinders is so proportioned that it represents 0·5 to 8 per cent. of the raw meal. The analysis of the dry pyrites cinders is: SiO_2, 1 to 3 per cent.; Fe_2O_3, 92 to 96 per cent.; SO_3, 1 to 3 per cent.

COKE BREEZE.—The bituminous coal coke breeze (size 0 to 10 mm.) is produced from Ruhr coal or Ruhr coke, and for a time Upper Silesian coke breeze was used. Analyses are as follows: Ruhr coke breeze—H_2O, 8 to 16 per cent.; C, 78 to 80 per cent. in dry state; ash, 10 to 15 per cent. in the dry state; the calorific value of the dry coke is 6800 to 7200 kg. cal. Coke breeze ash—SiO_2, 35 to 45 per cent.; Al_2O_3, 20 to 30 per cent.; Fe_2O_3, 15 to 20 per cent.; CaO, 6 to 10 per cent.; SO_3, 4 to 8 per cent.

PULVERISED BROWN COAL.—The pulverised brown coal has a residue of 15 to 20 per cent. on a 4,900-mesh sieve. Analyses are as follows: Brown coal dust—H_2O, 12 to 15 per cent.; C, 54 to 56 per cent.; ash, 10 to 12 per cent.; volatile constituents, about 40 per cent.; the calorific value is 4900 to 5100 kg. cal. Brown coal ash—SiO_2, 8 to 15 per cent.; Al_2O_3, 7 to 16 per cent.; Fe_2O_3, 2 to 3 per cent.; CaO, 25 to 45 per cent.; SO_3, 25 to 40 per cent.



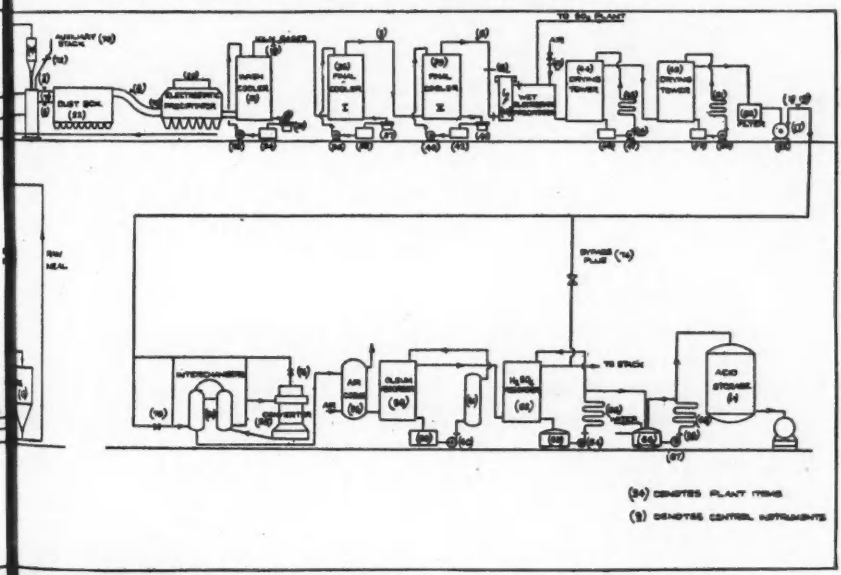
Flow-Sheet of Factory makiment

GYPSUM.—The gypsum, which is added at the cement grinding process, in the proportion of 2 to 3 per cent. of the clinker, has the following analysis: H_2O, 18 to 22 per cent.; $CaSO_4$, 76 to 82 per cent.; $CaCO_3$, 0 to 8 per cent.

RAW MEAL.—A typical raw meal is composed of approximately 78 per cent. anhydrite, 9·6 per cent. dry clay, 4·8 per cent. dry sand, 0·8 per cent. pyrites cinders, and 6·8 per cent. coke breeze. A typical analysis is as follows: H_2O, 0·5 to 1 per cent.; C, 4·5 to 5 per cent.; SiO_2, 8 to 10 per cent.; Al_2O_3 (including TiO_2), 3·5 to 4·5 per cent.; Fe_2O_3, 1·2 to 1·5 per cent.; CaO, 30 to 32 per cent.; SO_3, 45 to 47 per cent.; MgO, up to 0·2 per cent.; the "lime standard" is 96 to 100 per cent.; from 8 to 10 per cent. residue is left on the 4,900-mesh sieve. The "lime standard" is the ratio of CaO actually present to the theoretical equivalent of Fe_2O_3, Al_2O_3, and SiO_2 actually present.

The Kilns.

The raw meal is processed in four rotary kilns each 70 m. long by 3·2 m. in external diameter with a 20-cm. lining of high-grade firebrick. In the firing zone the kilns are lined for a length of 10 m. with magnesite bricks. The slope is 3·5 per cent. and the clinker is discharged through 12 tubular coolers. In the middle zone of the kiln three-armed firebrick bridges are built each 2 m. long, placed opposite each other. The total length of the bridges of one kiln is 14 to 22 m. In this manner the cylindrical space in the kiln is divided into three compartments and an improvement in the heat transfer between the gas and the raw meal is effected. The kiln makes one revolution in 90 seconds, and the material takes six to seven hours to pass through it.



ment and Sulphuric Acid.

Two products are formed during the treatment of the raw meal in the rotary kiln, namely, sulphur dioxide gas and clinker.

SULPHUR DIOXIDE GAS.—The raw meal passes into the kilns through a feed pipe by way of adjustable screw conveyors without granulation or moistening. The temperature at the upper end of the kiln is 520 to 600 deg. C. At about 900 deg. C. the reduction process commences and continues as the temperature rises, to be followed by true clinker burning in the firing zone where the temperature must be maintained at approximately 1,400 deg. C. The control of the kiln is effected by gas analysis ($CO_2 + SO_2$, O_2) at the kiln outlet by means of an Orsat apparatus and by adjustment of the quantities of brown coal dust burnt and of the quantities of gas drawn by the blowers through the kiln to the sulphuric acid plant. The process is so regulated that a very slight oxidising atmosphere exists in the kiln. Since in practice temporary reducing conditions, and consequently the formation of distillation products, cannot always be avoided, a blower is attached to the upper third of the kiln behind the bridges, to blow constantly a quantity of air into the kiln up to 5 per cent. of the raw gas. The investigators were told that secondary air was added so as to burn S and H_2S to SO_2. The secondary air blower is attached to the kiln and revolves with it blowing air into a box-ring.

With correct running of the kiln and at full load about 14,000 cubic metres of SO_2-containing gas are drawn out of the kiln. The composition of the gas (by volume) is 7·5 to 8·5 per cent. SO_2, 0·8 to 1 per cent. O_2, and 16 to 20 per cent. CO_2. This quantity corresponds to a daily output of about 100 tons of SO_3 per kiln. The gas leaving the top of the kiln at a temperature of about 520 to 600 deg. C. is passed through large capacity dust chambers of sheet iron with pear-shaped sections 12 m. long. The exit temperature is about 300 to 370 deg. C. The gas then passes through electrostatic dust precipitators with a cross sectional area of 7 to 9·4 sq. m. The exit temperature is about 220 to 300 deg. C. This cooling of the gases takes place by radiation. The flue gases were said to contain 80 to 90 gr. per cub. m. of H_2O and very little SO_3. A little NO was present and this appeared as 0·02 per cent. nitrogen oxides (measured as NO) in the final H_2SO_4 and also interfered with the absorption of SO_3. No trouble was experienced, with serious corrosion at any place in the dedusting system so long as the temperature remained about 300 deg. C.

CLINKER.—The clinker, after leaving the kiln and passing through the coolers, is still at about 250 deg. C. It has the following composition: 20 to 22 per cent. SiO_2, 8 to 9 per cent. Al_2O_3, 2·3 to 2·8 per cent. Fe_2O_3, 65 to 67 per cent. CaO, and up to about 2 per cent. SO_3.

At full load each rotary kiln has an output of about 250 tons per day of raw meal, which yields, in addition to about 100 tons of SO_3, about 120 to 125 tons of clinker. The consumption of coal dust corresponds to 1700 to 1800 kg. cal. per kg. of clinker.

CEMENT.—The cement, ground with the addition of 2 to 3 per cent. gypsum, corresponds to the standards for Portland cement " 225 " and represents a good

commercial grade. It has the following characteristics: Fineness—10 to 12 per cent. residue on a 4,900-mesh sieve; bending strength—at 7 days, 45 to 55 kg. per sq. cm.; at 28 days, 65 to 75 kg. per sq. cm.; compression strength—at 7 days, 220 to 280 kg. per sq. cm.; at 28 days, 400 to 460 kg. per sq. cm.

Manufacture of Sulphuric Acid and Oleum.

After leaving the dry electrostatic precipitator the gases flow to a tower in which they are cooled from about 300 deg. C. to about 60 deg. C. In this tower SO_2 containing water is circulated. The wash liquor runs through a wooden settling tank and six cooling coils, the cooled liquor being returned to the pumping tank. Since the wash liquor gradually becomes richer in chlorine, it is renewed as required. Leaving the tower the gases go to the first cooling tower where they are cooled to about 40 deg. C. by a spray. The final cooling of the gases is carried out in a second cooling tower where they are cooled to 22 to 35 deg. C. Final cleaning of the gas is done in wet electrostatic precipitators. The exit gases are optically clear. They are diluted with air before entering the driers in order to give the oxygen content necessary for the contact process, the quantity of air being such that the ratio of O_2 to SO_2 is at least 1·8. If it is not intended to convert the whole of the gases to sulphuric acid, a certain quantity of the gas can be passed to the SO_2 plant before dilution with air.

The gas drying process is in two stages. The cooled SO_2 gas, saturated with water vapour, passes through a drier, where the drying is effected with about 96 per cent. H_2SO_4. The sulphuric acid runs through a receiver, from which it is pumped to the drier through an irrigated cooler, which it leaves at about 45 deg. C. Final drying takes place in a second drier. The gas leaving the drier contains about 0·03 to 0·2 gm. per cubic metre of water. After leaving this drier the gas is ready for the contact process, that is it is clean, cool, dry, and contains the requisite amount of oxygen. Any acid mist entrained from the driers is trapped in a glass wool suction filter. The pumping of the gas is effected by blowers.

The contact gases, diluted with air after leaving the blower, have their air content adjusted to maintain the SO_2 content at about 4·5 to 5·5 per cent. A single unit treats the gas from one kiln and has two contact systems, each comprising a double heat-exchanger and one converter with three contact masses. The first and the third contact mass are in the form of beds, while the second is packed in tubes. Owing to the large surface of the exchangers it is possible, even when the SO_2 gases are temporarily weaker (down to about 3·0 per cent.), to maintain continuously the temperature necessary for the process. Vanadium catalyst of various sizes is used. Cylindrical particles of 10·6 mm. and 4 mm. diameter are used, with a V_2O_5 content of 30·5 gm. per litre in the case of the hard mass and of 45 gm. per litre in the case of medium hard mass.

The gases, containing about 5·0 per cent. of SO_2, enter the first heat exchanger from the bottom and pass into the second exchanger from top to bottom outside the tubes, while gases containing the SO_3 pass through the tubes in the opposite direction. The gases enter the heat exchanger at about 50 to 60 deg. C. and

leave at 280 to 320 deg. C. The temperature at the entrance to the first contact mass is about 490 deg. C. The gas leaves the first mass at a temperature of about 570 deg. C. (after about 80 per cent. conversion); the second mass is left at about 435 deg. C. (conversion about 95 per cent.) and finally the third mass is left at 445 deg. C. with a conversion of about 97·2 per cent. The gas containing SO_3 finally obtained is cooled to about 160 to 180 deg. C. in the heat exchanger before leaving the system.

The absorption of SO_3 can take place in the oleum absorber with a 98 per cent. absorber in series, or the whole of the contact gas can be led directly to the 98 per cent. absorber. For the production of oleum the SO_3 gas, at about 150 to 170 deg. C. is first cooled to about 60 deg. C. The cooled SO_3 gas (at about 60 deg. C.), then enters the oleum absorber. The oleum is pumped through a tubular cooler to the absorber. The temperature of the oleum at the tower inlet is about 45 deg. C., and upon leaving the tower returns to the pump at a temperature of 55 to 60 deg. C. Of the oleum in circulation a part is led off to storage, and this is continuously replaced by 98 per cent. absorber acid.

Sulphuric acid is produced in the sulphuric acid absorber. The spraying liquid, 98·5 per cent. H_2SO_4, is pumped to the absorber by way of a spray cooler at a temperature of 60 deg. C. The acid is maintained at the desired concentration by the addition of 96 per cent. acid from the drying system or by the addition of water to the 98 per cent. pump tank. Part of the acid produced is passed to the drying system for maintaining a concentration of 96 per cent. acid, while the surplus is passed directly to the sulphuric acid store or is similarly transferred to storage by way of the diluting plant. The sulphuric acid produced has an F. content of 0·001 to 0·003 per cent. It is free from arsenic but contains about 0·02 per cent. nitrogen oxides (measured as NO), the formation of which in the rotary kiln during combustion of the brown coal is unavoidable. Owing to the presence of the nitrogen oxides a small proportion of the SO_3 passes unabsorbed through the stack with the exit gases. The acid, when it is not sold as 98 per cent. acid, is adjusted to the required strength with water in a small diluting plant.

The sulphuric acid and oleum are stored in tanks placed at such a height that the acid can be directly siphoned into tank wagons. The storage tank for oleum of a strength exceeding 20 per cent. is isolated and the base is provided with a false bottom, which is kept at the required temperature with steam heating coils.

Operation of Plant.

The rotary kiln is heated with producer gas. As soon as the walls are glowing the kiln is turned through 90 deg. every two hours. When the kiln has reached 800 to 1,000 deg. C. (this takes about half a day), pulverised coal, which ignites immediately, is injected and the kiln is then continuously rotated at the minimum speed of one revolution every 2½ minutes. The temperature in the kiln rises rapidly if the oxygen content in the kiln head does not appreciably exceed 1 per cent. The raw meal feed is approximately 6 tons per hour when the temperature in the firing zone is about 1,300 deg. C. and that in the kiln head is from 400 to 450 deg. C. After 2½ to 3 hours the gas analysis indicates that

reaction has commenced, while the firing zone temperature has risen to 1,330 to 1,400 deg. C. The oxygen content in the kiln head is kept at about 0·5 per cent. When the firing zone temperature reaches 1,400 deg. C. the producer gas flames are extinguished. The kiln is then run at a speed of one revolution every 1½ to 2 minutes until the reaction is proceeding satisfactorily, which is usually after 5 to 8 hours. After this period the first clinker is discharged although not yet fully burned. Meanwhile the blanks between the kiln head and the dedusting chambers have been taken out but the dampers at the inlet of the electrostatic precipitators still remain closed.

When the kiln is steadily evolving SO_2 the change-over to the blower or contact system is effected. The change-over of the gas from the auxiliary stack to the gas blower is carried out as follows. The dampers at the inlet of the electrostatic precipitators are opened and the blower is started. The gas stream does not pass into the contact system but is blown through a by-pass damper directly into the flue and thence into the chimney. The damper in the auxiliary stack is then closed. The kiln is adjusted to an oxygen content in the gas of 0·5 to 1 per cent. by means of the blower or by regulating the pulverised coal feed. As soon as conditions are steady and a sufficient SO_2 concentration is obtained on the pressure side of the blower, the secondary air blower on the kiln is started and the gas is adjusted to the correct content of SO_2 and O_2 by altering the openings on the secondary air supply. When the SO_2 and O_2 contents are 4 per cent. and 8 per cent. respectively, the by-pass damper to the flue is slowly closed and the inlet valve of the previously heated contact unit is opened. The electrostatic precipitators are switched in as soon as the inlet temperatures have risen to about 200 deg. C. The kiln is gradually brought to the maximum output of raw gas by increasing the quantity of raw meal and pulverised coal and by adjusting the gas blower; this takes less than 24 hours.

Meanwhile the catalyst system is being heated. The contact system is sufficiently hot and ready for operation in five to six days when the temperature at the inlet to the first contact mass is from 480 to 500 deg. C.

Examination of Samples.

The following samples were brought back from Wolfen: 1 kg. of anhydrite, 1 kg. of raw meal, 1 kg. of cement, 1 piece of magnesite brick, 2 pieces of firebrick, 1 kg. of vanadium catalyst. Some of these have been examined by I.C.I. (Billingham Division).

The anhydrite was not analysed, as it was considered that the I.G. Farbenfabrik analyses were adequate.

The sample of raw meal was reduced to 10 gm. in a moving hopper sampler and a complete size analysis was done on the 10-gm. sample by sieve grading down to a 240-mesh B.S. sieve. The size analysis of material on the sub-sieve range was determined in two parts: (1) Larger than the range 66–20 μ using the Richardson turbidimeter. (2) Smaller than 20 μ by elutriation. The results are given overleaf.

Sizes	Percentage between sizes
+ 72 B.S. sieve	1·4
− 72 + 170 B.S. sieve	4·9
− 170 + 240 „	7·1
Sub-sieve analysis particle size	
76·0 to 53·0 microns	—
53·0 to 37·5 „	14·1
37·5 to 18·8 „	24·1
Smaller than 18·8	48·4

The I.G. report gives a grading of 8 to 10 per cent. residue on a 4,900-mesh sieve, which is approximately equivalent to a No. 170 B.S. sieve; the I.C.I. grading shows about 6·3 per cent. residue on this sieve.

The chemical analysis compared with the I.G. analysis is as follows:

	I.C.I. analysis	I.G. analysis
SO_3	43·5	45 to 47
CaO	32·2	30 to 32
SiO_2	9·8	8 to 10
Al_2O_3	3·0	3·5 to 4·5
TiO_2	0·3	
Fe_2O_3	1·0	1·2 to 1·5
Free C	5·2	4·5 to 5·0
Combined H_2O	2·5	0·5 to 1·0
CO_2	1·2	—
MgO	0·4	up to 0·2
K_2O	0·3	—
Na_2O	less 0·1	—
P_2O_5 and Cl	than 0·1	—
Sulphide	„ 0·1	—
As	„ 2 parts per million	—
	99·4	

The chief results of the cement tests are as follows:

	Sample	I.G. figures	B.S. specification
	per cent.	per cent.	per cent.
SO_3	3·55	up to 2	2·75
Sulphide	0·35	—	—
Free lime	1·92	—	—
Combined water	0·43	—	—

The SO_3, sulphide, and free lime contents are all high and suggest an incomplete kiln reaction for this sample. The combined water content corresponds with 2·06 per cent. gypsum ($CaSO_4\ 2H_2O$), of which 0·96 per cent. is SO_3; this agrees with the I.G. statement that 2 to 3 per cent. gypsum is added to the clinker. The cement met the British Standard requirement for soundness, the Le Chatelier expansion being 2 mm., but it was noticed that a hair crack developed on the

pat in the curing period before boiling. Compressive strength determinations were carried out, using ½-in. cubes. Apart from having somewhat low strength at one day, the strength development was satisfactory. It can be concluded that the sample was not a very good cement, but the sample was probably not typical of that which can be obtained by this process.

The tests suggested that the magnesite bricks were of good quality, but no better than the best British equivalents. There appeared to be nothing outstanding about the firebrick, and no examination was carried out.

The catalytic activity of the sample of vanadium catalyst was tested under standard laboratory conditions over a temperature range of 410 to 480 deg. C. Two small samples were selected at random from the sample provided to give an indication of the homogeneity of the catalyst. Activity coefficients were calculated according to the method described in Z.f.Physik. Chem. 1931, 30 (G. B. Taylor and S. Lenher).

Lurgi Catalyst

Sample No. 1		Sample No. 2	
Temperature (deg. C.)	Activity coefficient	Temperature (deg. C.)	Activity coefficient
416	4·7	416	4·7
433	50·0	438	14·2
449	80·0	457	40·0
477	93·0	480	85·0

The Wolfen catalyst is apparently not homogeneous, but nevertheless the activity appears to be normal. The activity determinations were carried out at atmospheric pressure and under nearly isothermal conditions. Space velocities were not corrected for variation of gas volume with temperature, and this factor is therefore included in the temperature variation of the activity coefficient. The catalyst, which had been made by extrusion, 4 mm. dia., contained 8·9 per cent. V_2O_5 and presumably was very similar to the catalyst made at Ludwigshafen and described in B.I.O.S. Report 244.

The Supply of Bags for Lime.

Speaking at a recent meeting of the Southern Lime Association, the chairman, Major E. W. Taylerson, said that the delivery of hydrated lime was becoming extremely difficult owing to a shortage of paper bags. The chalk and lime industry at present required over 3,000,000 paper bags per annum, and with the growing demand for lime for building this would undoubtedly increase during 1947. He pointed out that although the Ministry of Supply refused to allow more paper to be used for this purpose, other Ministries were requiring lime.

The Combination of Oxides in Portland Cement.

A STUDY of a portion of the system Na_2O-CaO-Al_2O_3-SiO_2 has been made by Messrs. Kenneth T. Greene and R. H. Bogue as a step in the solution of the problem of the state of combination of Na_2O in Portland cement clinker, and the results are given in a paper entitled " Phase Equilibrium Relations in a Portion of the System Na_2O-CaO-Al_2O_3-SiO_2," issued by the U.S. National Bureau of Standards.

In an introduction the authors state that in order to understand the state of chemical combination of the various component oxides of Portland cement it is necessary first to study the interrelations of these oxides as systems of few components, gradually extending the scope of the investigations to include other oxides and, consequently, more complicated systems, as more and more data become available. The phase equilibrium relations of the major oxides of Portland cement, CaO, SiO_2, Al_2O_3, Fe_2O_3, and MgO have been rather thoroughly determined and the results are well known. However, it is only within the last few years that a systematic attempt has been made to study the effect of such minor components as SO_3, K_2O, Na_2O, TiO_2, etc., upon the constitution of Portland cement clinker. This paper represents part of an investigation undertaken to determine the manner in which one of these minor components, Na_2O, combines with the other oxides of clinker.

The results of the investigation are limited in their application to commercial Portland cement by the fact that one of the major components of clinker, Fe_2O_3, has been excluded. Furthermore, the exact effect of a number of the minor components is not yet known. Nevertheless, on the basis of present information, the authors state that it is possible to reach a much more definite conclusion than heretofore regarding the state of the Na_2O in clinker. Several possibilities are indicated, depending not only on the composition of the clinker but perhaps even more on the heat treatment which it receives.

If cooling has been rapid so that glass is present in the clinker, some of the Na_2O will always be found in this glass. The percentage of Na_2O occurring in this manner will depend upon different conditions, but some indication may be obtained from the phase equilibrium data for the Na_2O-CaO-Al_2O_3-SiO_2 system. For example, a glass resulting from the quenching of a liquid near in composition to that of point 1 contains approximately 3·5 per cent. Na_2O. However, if this melt is allowed to crystallize independently until the composition of the remaining liquid approaches that of point 5, and then is quenched, the resulting glass will contain upwards of 8 per cent. Na_2O.

The results thus far obtained indicate that, when the cooling takes place at a slower rate, some of the Na_2O in a clinker may crystallize as the compound Na_2O. 8CaO.3Al_2O_3, or more likely as a solid solution phase of this compound and 3CaO.Al_2O_3. It is probable that this phase is closely related to the " prismatic dark interstitial " material found in some commercial Portland

cement clinkers. The compound $Na_2O.8CaO.3Al_2O_3$ has been observed in the present work to form prismatic crystals when the cooling rate was suitable, and it has been shown that the dark prismatic phase of clinker is closely related to $3CaO.Al_2O_3$. Although no proportionality was found between the percentage of alkali in a clinker and the amount of the dark prismatic phase which may be produced, the presence of either Na_2O or K_2O appears to be necessary for its formation.

The fact that $2CaO.SiO_2$ can form solid solutions containing Na_2O makes this another possible manner of occurrence in clinker. The opinion has been expressed that, because of their instability, the α-$2CaO.SiO_2$ solid solutions which may be formed at clinkering temperatures are probably not present in normally-cooled clinker. However, since the material held in solid solution at the higher temperature is precipitated as inclusions in the $2CaO.SiO_2$ grains when they invert to the β form, some of the Na_2O will be contained in these inclusions. This fact may be of importance in the hydration of cement, since β-$2CaO.SiO_2$ hydrates slowly and the alkali embedded in the grains might be protected from the action of the water for considerable lengths of time.

The addition of Na_2O to the system CaO-Al_2O_3-SiO_2 was found to result in an enlargement of the primary phase region of CaO. This means that certain compositions in the alkali-free system would not contain free CaO at clinkering temperatures, but after addition of Na_2O would contain stable CaO. In this way Na_2O can affect adversely the combination of the CaO in the cement mix.

The authors give the following summary of the paper. The portion of the quaternary system Na_2O-CaO-Al_2O_3-SiO_2 of importance to Portland cement technology has been studied by the quenching method. The phase equilibrium data are presented in the form of tables and phase diagrams, and features of interest are discussed. Mixtures located in three definite composition planes passing through the four-component tetrahedron, as well as a number of additional compositions, were investigated. Twelve crystalline compounds were observed to have primary phase volumes within this portion of the system. Five of the quaternary invariant points occurring in this part of the system have been located approximately. Of these only one is a eutectic; the other four are reaction points. It was found that $2CaO.SiO_2$, $3CaO.SiO_2$, $3CaO.Al_2O_3$, and $Na_2O.8CaO.3Al_2O_3$ are capable of coexisting with liquid at one of these reaction points. The temperature of this point is 1,440 deg. $\pm$ 10 deg. C. and the composition is 3·5 per cent. Na_2O, 55·2 per cent. CaO, 31·0 per cent. Al_2O_3, and 10·3 per cent. SiO_2. Evidence was obtained indicating that, at temperatures below that of liquid formation, $3CaO.Al_2O_3$ and $Na_2O.8CaO.3Al_2O_3$ may combine under suitable conditions to form a solid solution phase of intermediate properties and crystal structure. Based on present information, and ignoring the effect of Fe_2O_3 and minor components, it is concluded that the Na_2O in Portland cement clinker may occur in several ways depending on different conditions: (1) As a component of glass; (2) As $Na_2O.8CaO.3Al_2O_3$ or a solid solution of that compound

with $3CaO.Al_2O_3$; such a phase may be associated with the prismatic dark interstitial material frequently observed in clinker; (3) As a solid solution of a soda-bearing phase in $2CaO.SiO_2$; and (4) As inclusions of a soda-bearing phase in β-$2CaO.SiO_2$ produced by ex-solution on inversion from α-$2CaO.SiO_2$.

Screen Analysis of Coal.

ACCURATE analyses of the grading of coal, such as are required for performance and efficiency tests of industrial plants, are difficult to obtain since the size and source of the sample and the method of screening are among the many factors that affect the result. A tentative specification, B.S.1293 of 1946, issued by the British Standards Institution, has therefore been prepared with the object of obtaining from the examination of a sample of coal, other than pulverised coal, a screen analysis which within reasonably definite limits of accuracy is representative of the whole consignment. Observations relating to coals of the sizes used in cement works in Britain are given in the following notes which are based on this standard.

Collection of Samples.

The place at which samples are obtained is dependent on the purpose for which the test is to be made, and if samples are taken at the consumers' works they may be collected at any stage between the unloading of the coal and feeding it into the kilns or boilers. Care must be taken to ensure that samples are not liable to be unrepresentative due to separation. It is, however, preferable for samples to be taken while the coal is in motion and they should be taken by an experienced person.

If the samples are taken from the discharge ends of chutes or conveyors, successive quantities should be obtained at regular intervals by plunging a ladle into the falling stream of coal. Each quantity so obtained should be taken from the full width and thickness of the stream, but, where this is impracticable, quantities should be systematically collected from all portions of the stream. The ladle should not be filled to overflowing and should be plunged into the stream from the back and the front alternately. Where the samples are collected from conveyor-belts the coal should be taken at regular intervals from the complete width of the belt, as considerable separation occurs on conveyors. Where such collection is impracticable quantities should be taken from each side and the middle of the belt in such a way as to counteract the effect of separation and uneven loading of the belt. When samples are collected from bucket elevators, the complete contents of a bucket should be taken at regular intervals.

If the samples are taken from a truck, the collection should be made while unloading the truck by taking the whole contents of the unloader's shovel at regular intervals throughout the entire period of unloading. It is difficult to obtain characteristic samples from heaps of coal, except from small piles not exceeding 3 ft. in height. In the latter case a fairly accurate sample can be obtained by taking a number of samples from points evenly distributed throughout the heap.

Weight of Sample.

The gross weight of the sample obtained by mixing together all the separate quantities collected should not be less than 75 lb. for coal of 1½-in. maximum size and 50 lb. for coal of 1-in. or under. Each gross sample should be composed of at least 25 separately collected quantities, the minimum weight of each being 3 lb. for 1½-in. coal and 2 lb. for coal 1-in. and under. In the case of ½-in. coal the gross sample may be reduced to 10 lb., and this should be done by riffling and not by quartering as the latter method leads to errors caused by separation.

Screening.

Before it is screened the sample should generally be dried. Coals containing fines should be air-dried. The screen should have square apertures, and the use of the following sizes will generally prove satisfactory: ⅛ in., ¼ in., ⅜ in., ½ in., ¾ in., 1 in. and 1½ in. Where screens with round holes are used, the sizes can be converted to equivalent square holes by dividing by 1·16, this factor being applicable to British coal of small size.

The samples of coal and the fractions after screening should be weighed within an accuracy of 0·5 per cent., and the sum of the weights of the fractions should be within 0·5 per cent. of the weight of the original sample.

The sample should be screened in such quantities as will allow each piece of coal retained on the screen to be in direct contact with the screen upon completion of the screening operation. Where the material passing the screen has to be re-screened, the coal should not be allowed to fall more than 6 in. For coal not exceeding 1½ in. in size, the screen should be moved horizontally at just sufficient speed to cause the pieces of coal to roll on the screen, and the motion of each particle should not be stopped by impact on the sides. The standard method of screening requires a cycle of four horizontal movements in each direction followed by two upward jerks. This cycle is repeated four times. The coal is passed through the largest screen first.

Alternatively coal not exceeding 1½ in. in size can be screened on mechanical shaker screens inclined at an angle of 13 deg. and receiving 190 upward movements per minute. In this case the coal passes over the finer mesh first.

Accuracy.

The accuracy of the foregoing method of analysis depends on the error involved in the collection of the sample and the screening. The degree of accuracy varies with the percentage of material retained between two successive sizes of screens, the accuracy being higher when the percentage is very small or very high. The greatest inaccuracy in sampling occurs when the amount of material retained between two successive screens is 50 per cent. of the sample. Thus, in selecting the sizes of the screens, it is preferable that the weight of coal retained between two successive sizes of screens should not be between 30 per cent. and 70 per cent. of the weight of the sample. Other inaccuracies occur due to the movement of the coal, causing breakages which alter the particle distribution.

If sampling and screening of coal under 3 in. is performed as described, in

nine cases out of ten the limits of deviation from the true value will not exceed the following :

Percentage between two screens	Sampling deviation (per cent.)	Screening deviation (per cent.)	Total deviation (per cent.)
2 or 98	1·0	0·50	1.10
5 or 95	1·5	1·00	1·80
10 or 90	2·0	1·00	2·25
20 or 80	2·0	2·00	2·80
25 or 75	2·5	2·50	3·50
30 or 70	3·0	2·50	3·90
40 or 60	4·0	2·75	4·90
50	5·0	3·00	5·80

This standard of accuracy means that if, for example, the true weight of the fraction of the coal lying between two screen sizes is 30 per cent. of the weight of the sample, then in one case out of ten the result of the screen analysis will be equal to or less than $30 - 3{\cdot}9 = 26{\cdot}1$ per cent., or will be equal to or greater than $30 + 3{\cdot}9 = 33{\cdot}9$ per cent. In the other nine cases the result will be nearer the true value.

If greater accuracy is required, duplicate samples should be obtained and the mean of the two results taken. Alternatively a sample of greater weight than is theoretically required should be obtained and analysed. The recommended minimum weights of the samples are based on the weights theoretically required to maintain the sampling deviations within the figures given in the table. These theoretical weights are 70 lb. for 1½-in. coal, 25 lb. for 1-in. coal, and 5 lb. for ½-in. coal, compared with the recommended minimum weights of 75 lb., 50 lb., and 10 lb. respectively. The reason for the increase over the theoretical weights is that, with less than 25 collections in each sample, the latter is not representative. Doubling the theoretical quantity of the sample reduces the sampling deviations to $\sqrt{2}$ of the tabulated values, with a consequent reduction in the total deviation, and similarly for other degrees of increase. This part of the standard is based on the researches of Dr. Manning on the theory of sampling granular material for the determination of size distribution.

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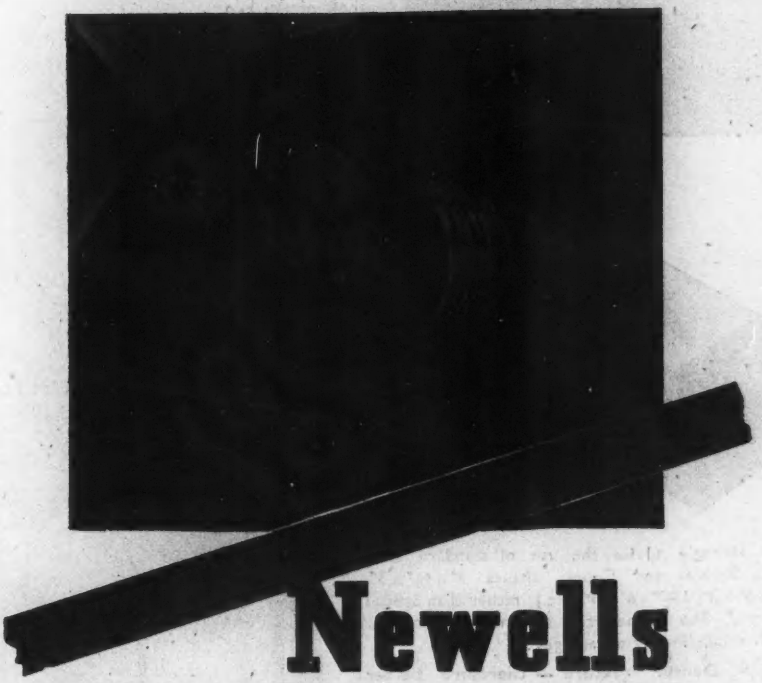

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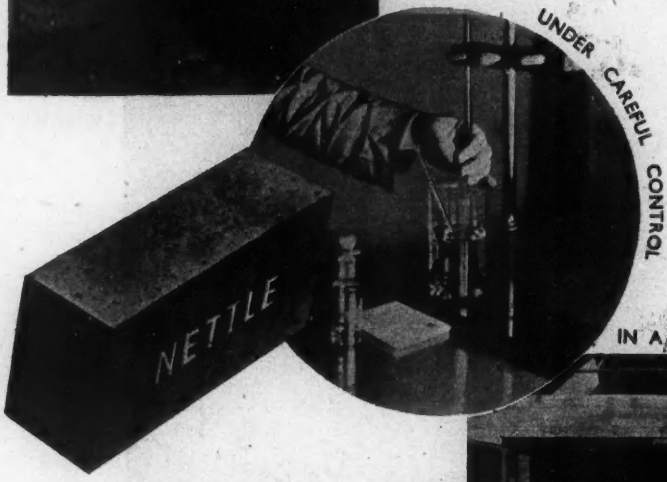

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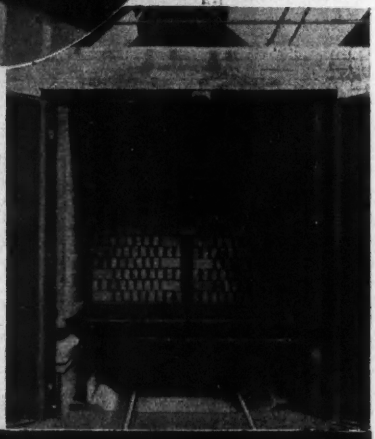

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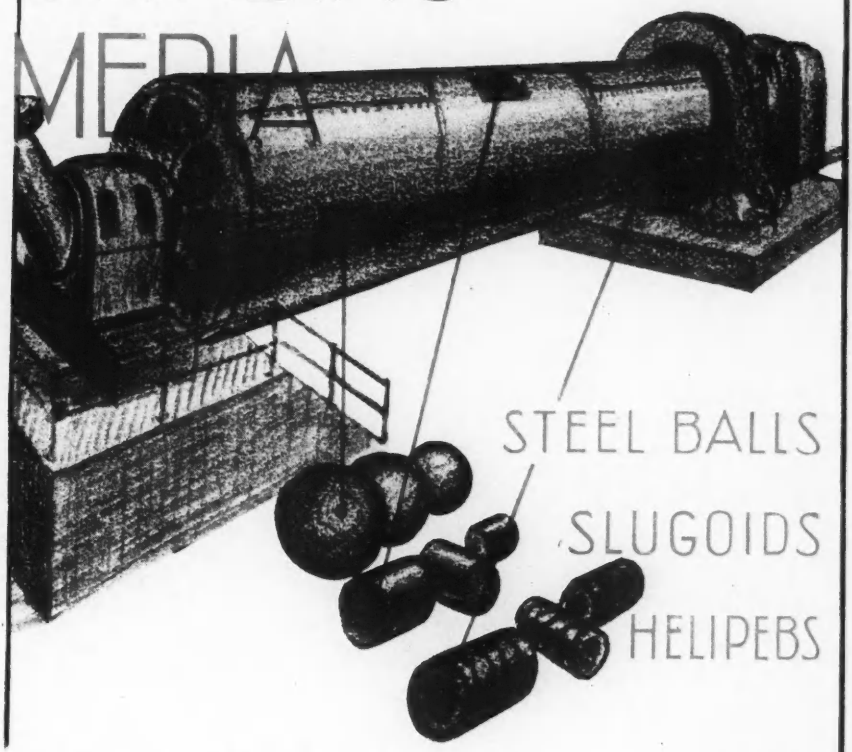

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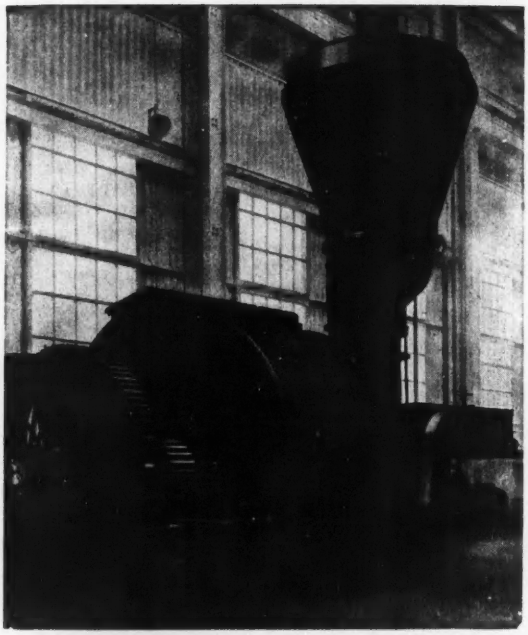

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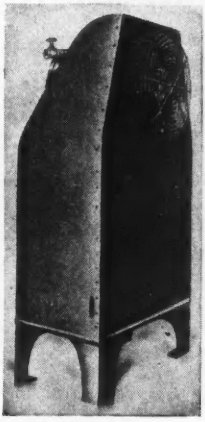

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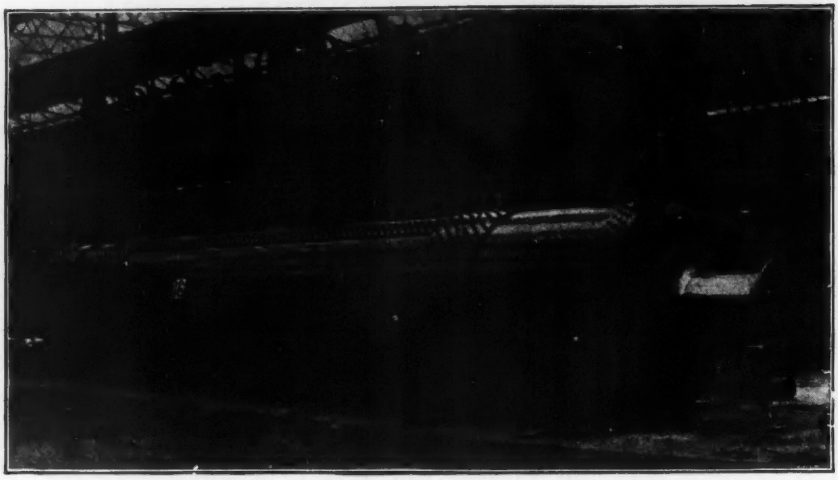

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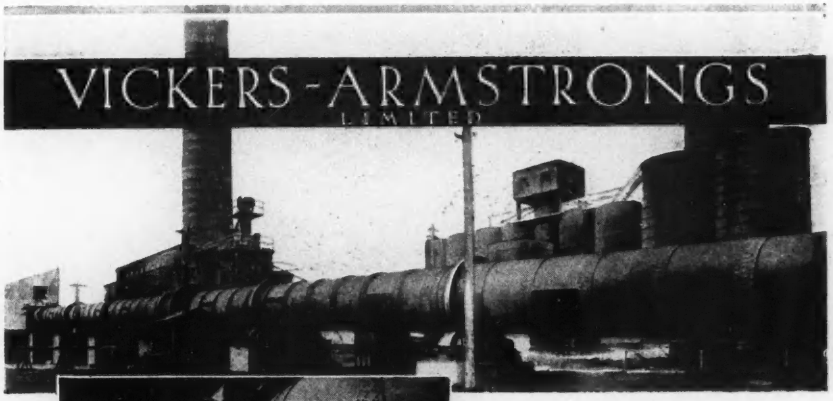

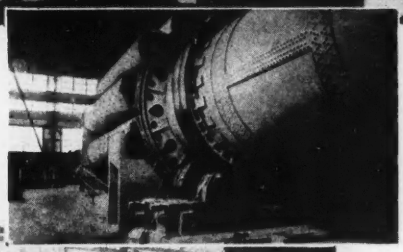

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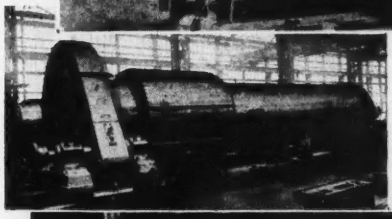

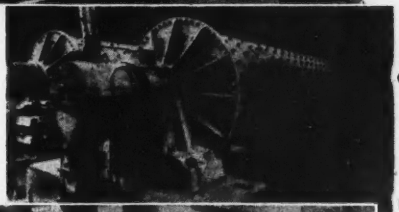

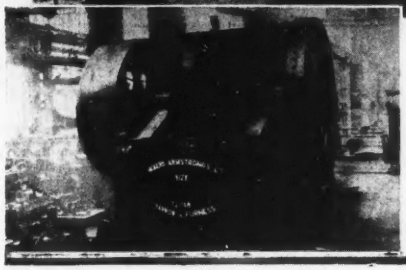

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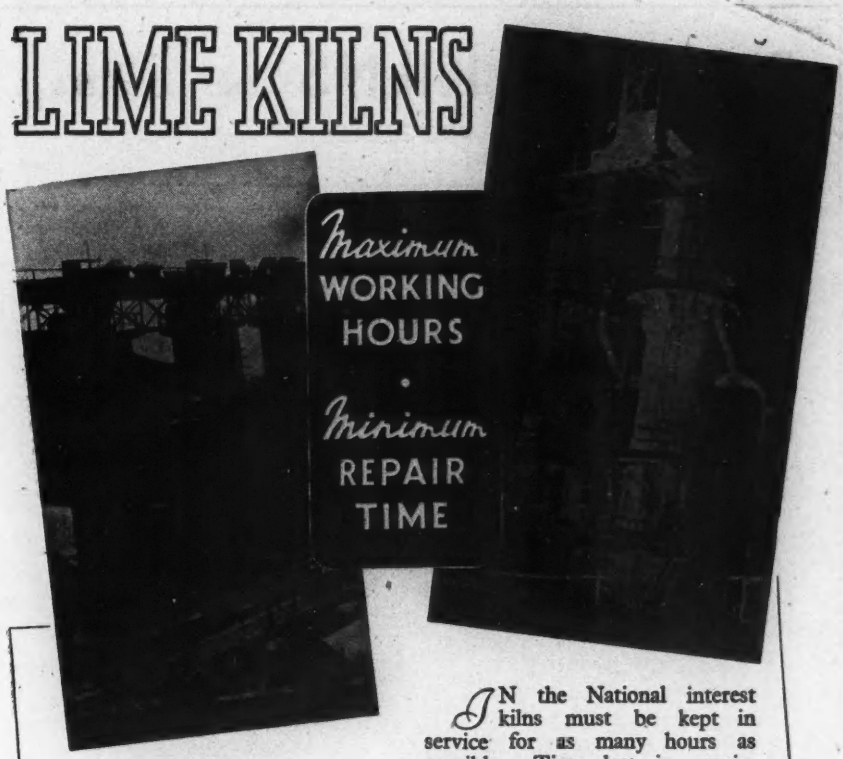

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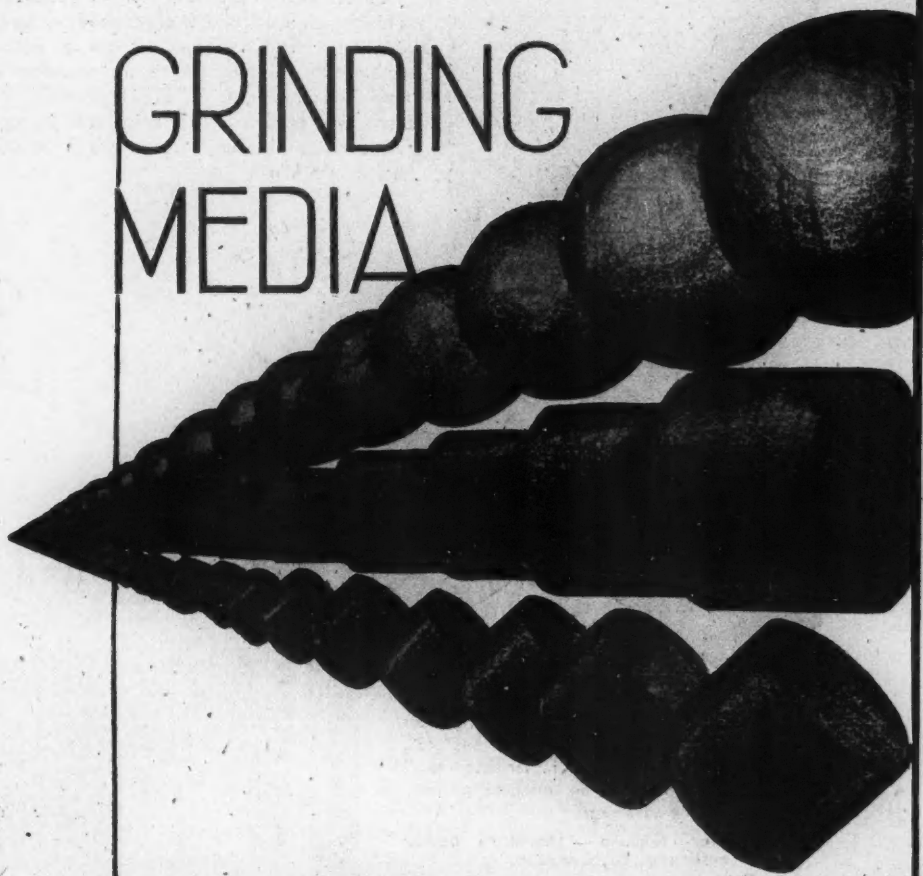

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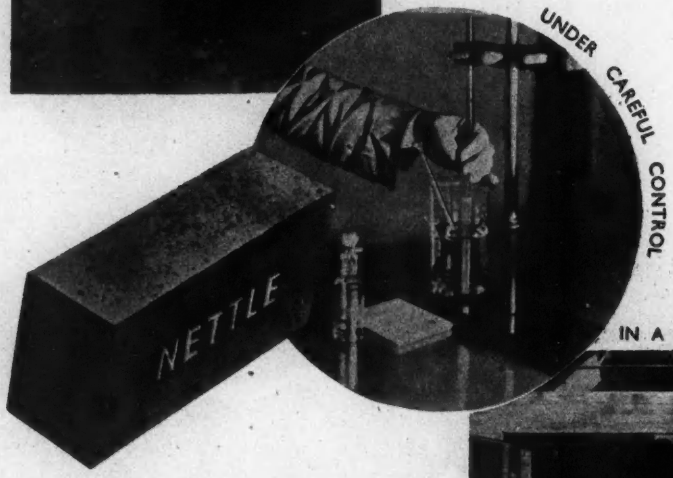

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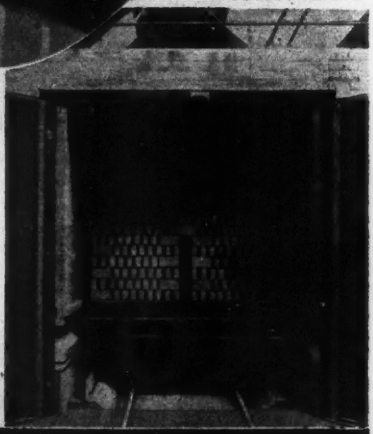

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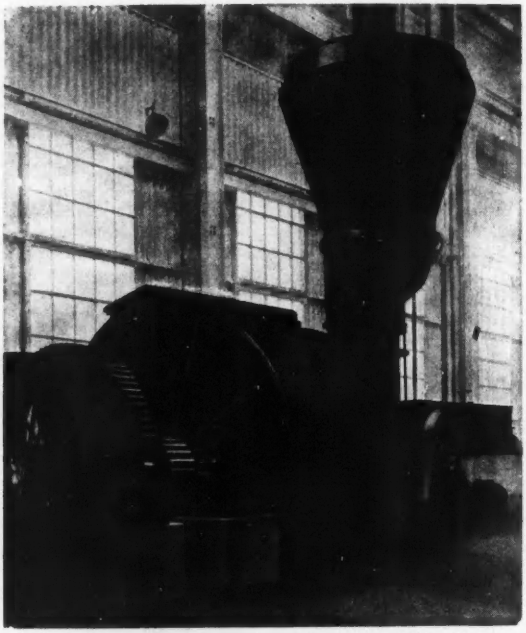

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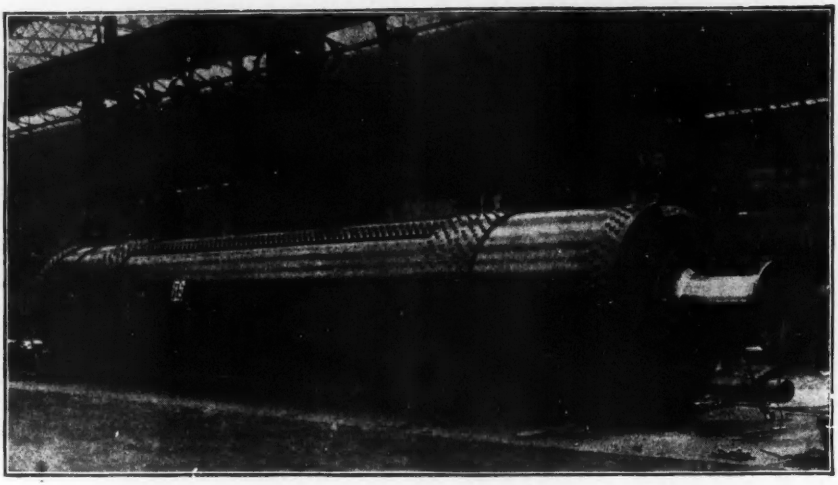

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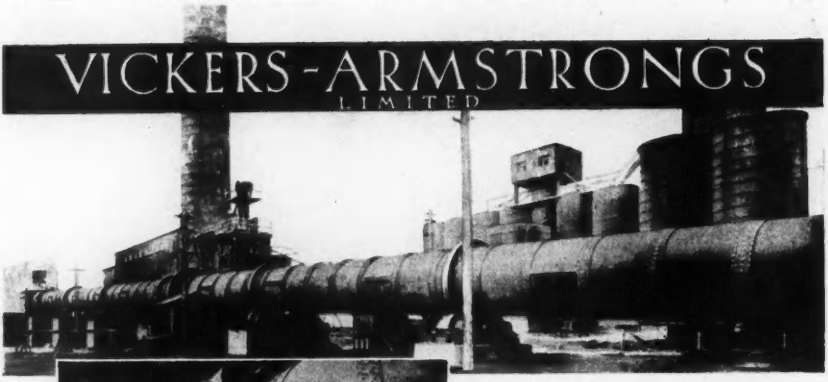

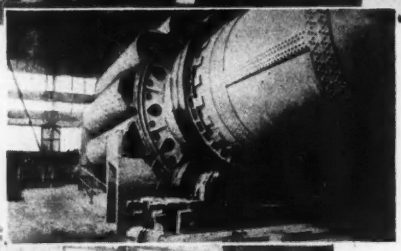

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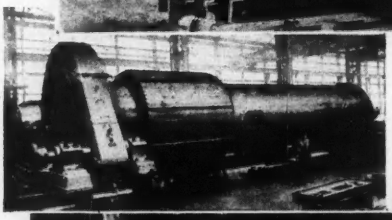

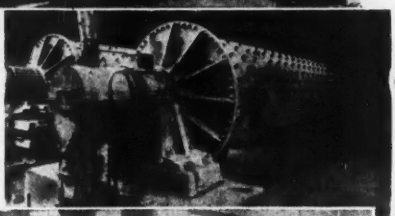

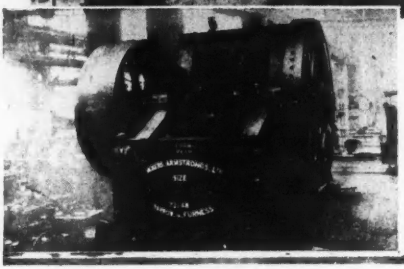

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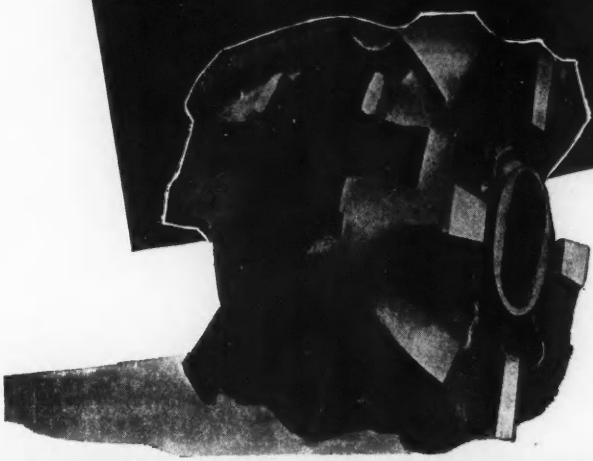

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is sometimes the price of a Ransomes Electric Truck. To be more specific, there are cases where a Ransomes Electric Truck has saved its first cost in six months in the economies effected in time and labour.

There are sizes and types to suit every job, 1-, 2- and 4-tonners.

The illustration is of the famous 1-tonner.

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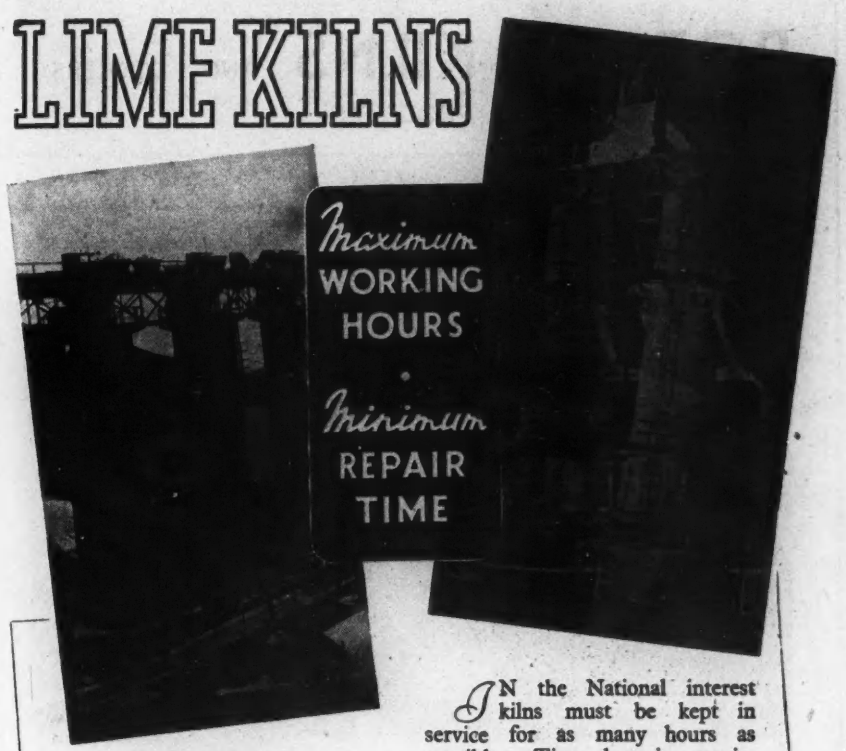

ADAMANTINE

Blue Adamantine blocks and bricks are hard-burnt, tough, and offer great resistance to abrasive action. They make excellent close-jointed, firm-faced brickwork which will withstand enormous pressure, and are suitable for use in the top and bottom zones.

HYSILYN

Hysilyn lime-resisting refractories, for use in the calcining zone, possess great mechanical strength in addition to the ability to withstand severe temperatures and alkaline attack. This quality is supplied with a content of alumina varied to meet the requirements of specific working conditions.

IN the National interest kilns must be kept in service for as many hours as possible. Time lost in repairs must be minimised. The reputation achieved by Davison Refractories in all parts of the world for over one hundred years guarantees their thorough dependability.

These Refractories are supplied in shapes to suit any type of construction including the Davison tongued and grooved system which adds considerably to the strength of any lining.

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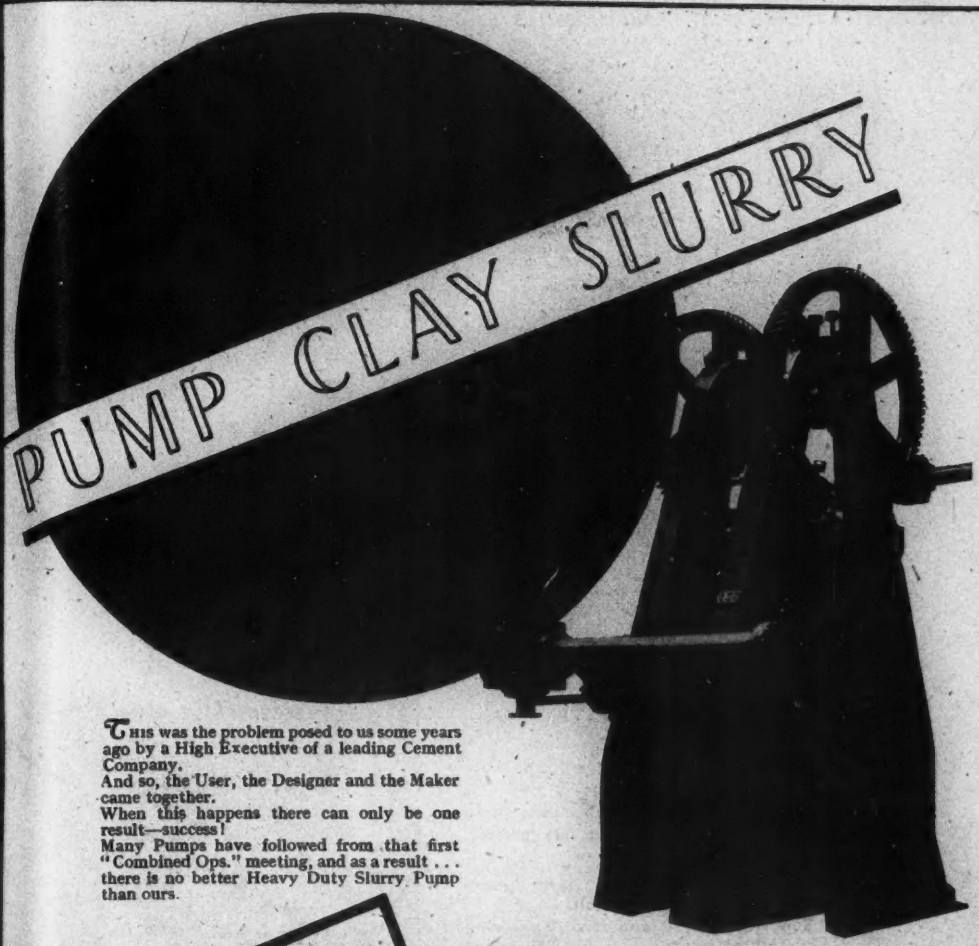
JUN 11 1946

CEMENT AND LIME MANUFACTURE

Vol. XIX. No. 3 MAY 1946 PRICE 1/- ALTERNATE MONTHS

PUMP CLAY SLURRY



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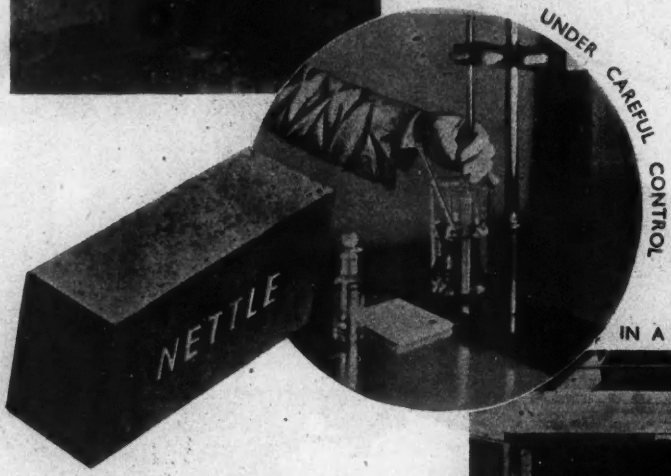
"NETTLE" BRAND FIREBRICK (42/44% Alumina) is highly refractory and displays good resistance to abrasion and corrosion. These properties enable it to withstand the severe conditions in the Calcining Zone. "STEIN GLASGOW" Brand is a hard, dense firebrick and its good resistance to abrasion recommends it for the shaft of the kiln.



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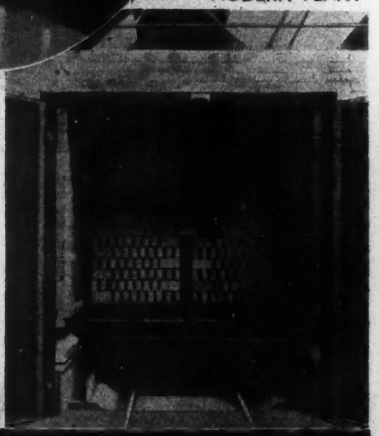

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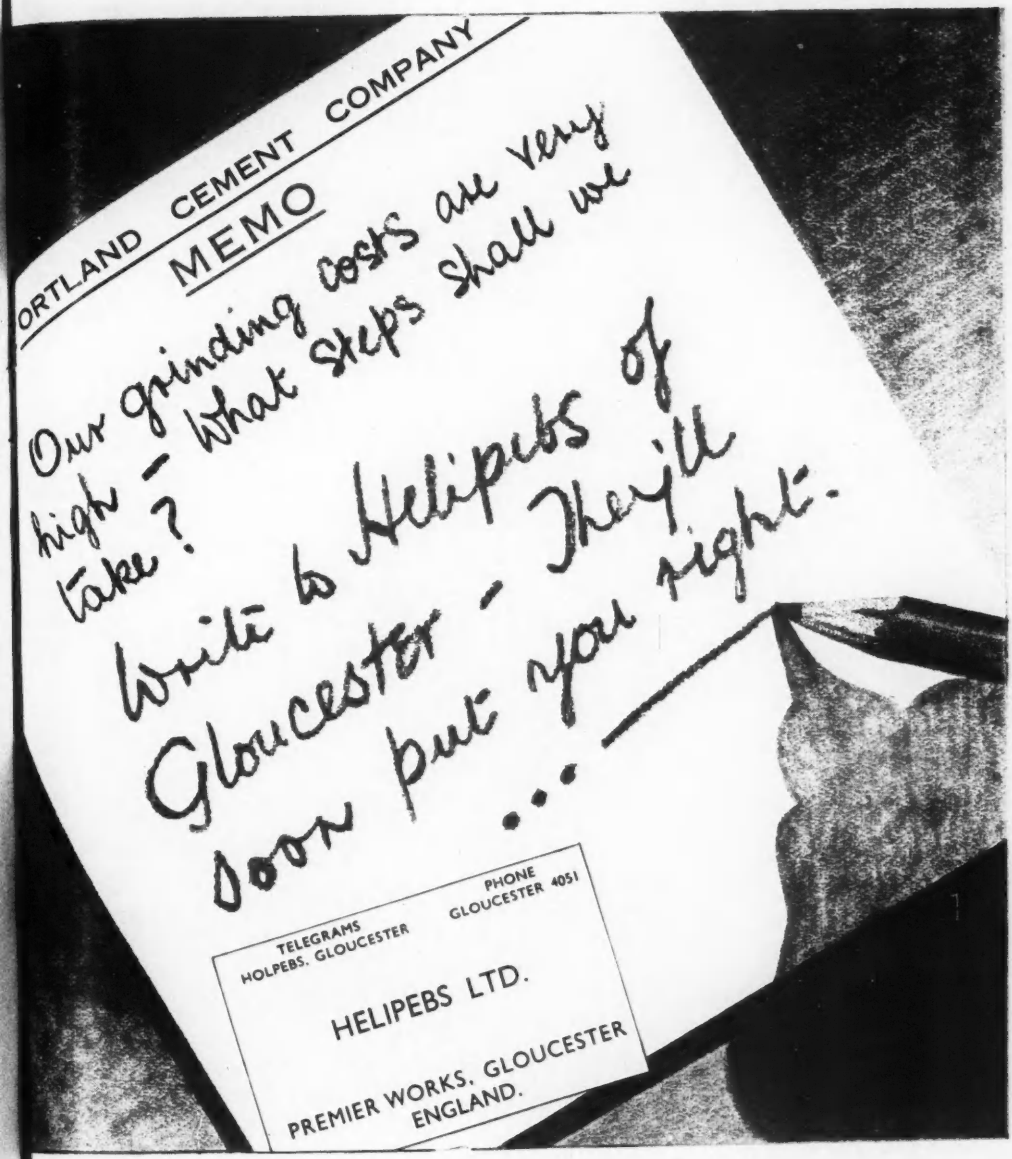
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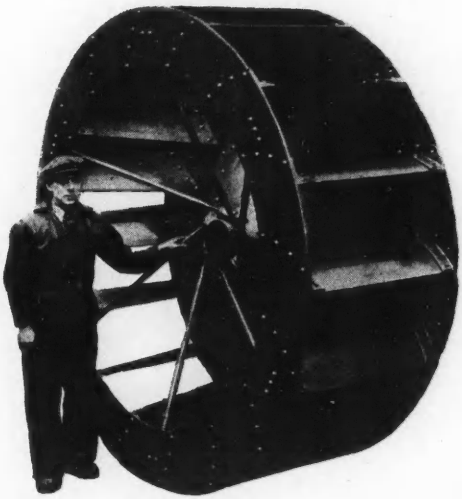

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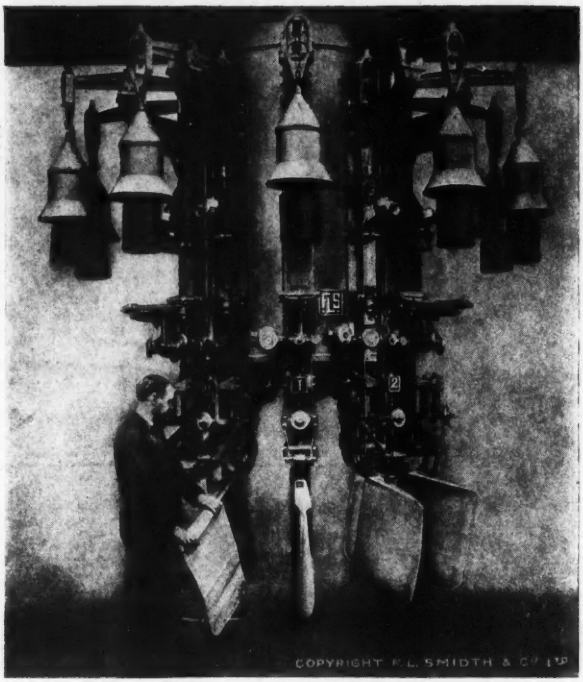

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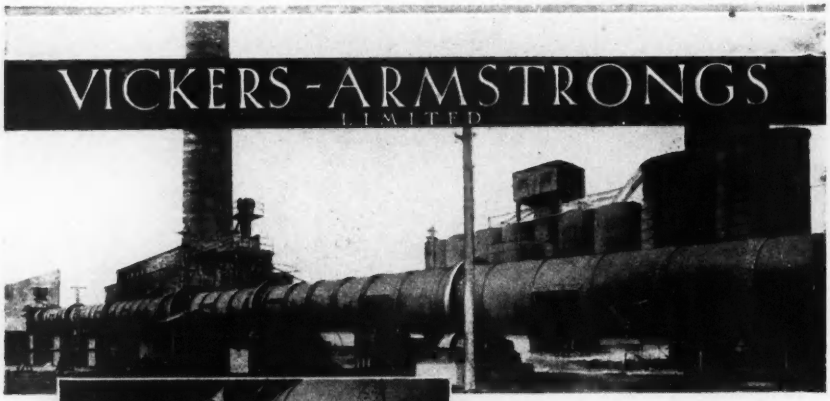

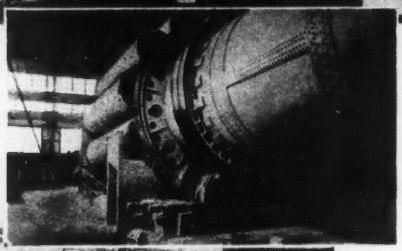

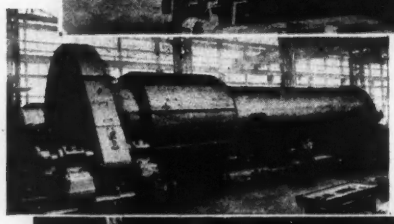

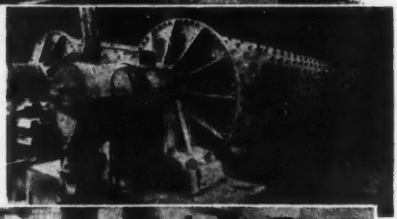

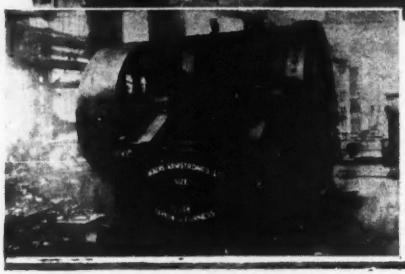

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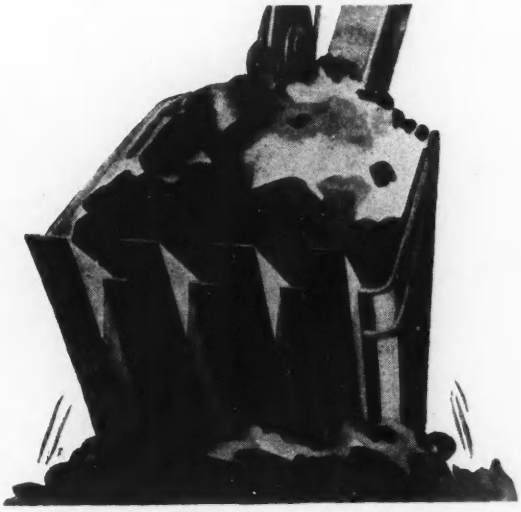

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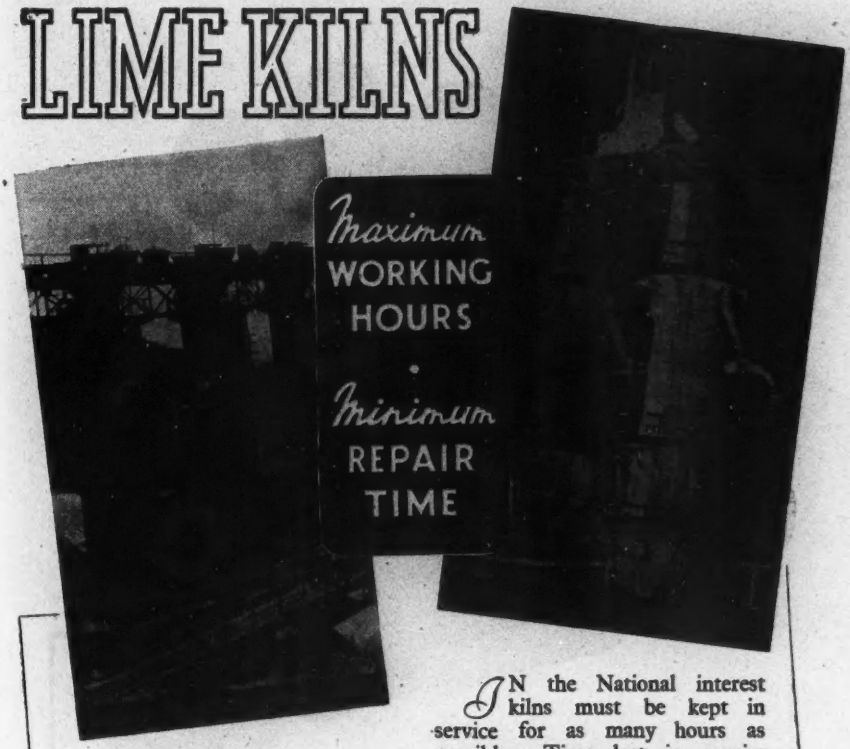

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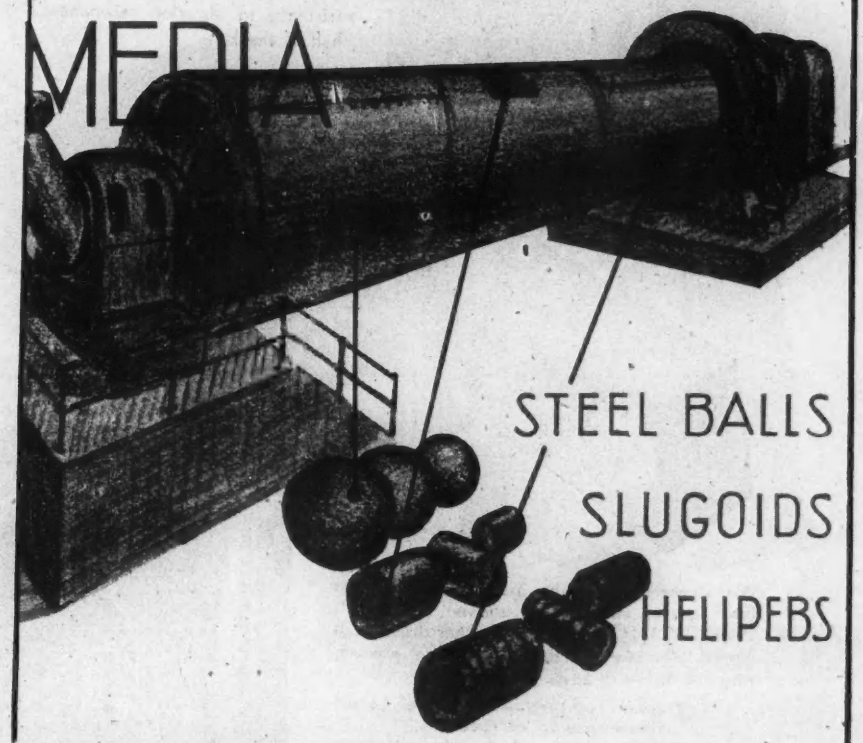

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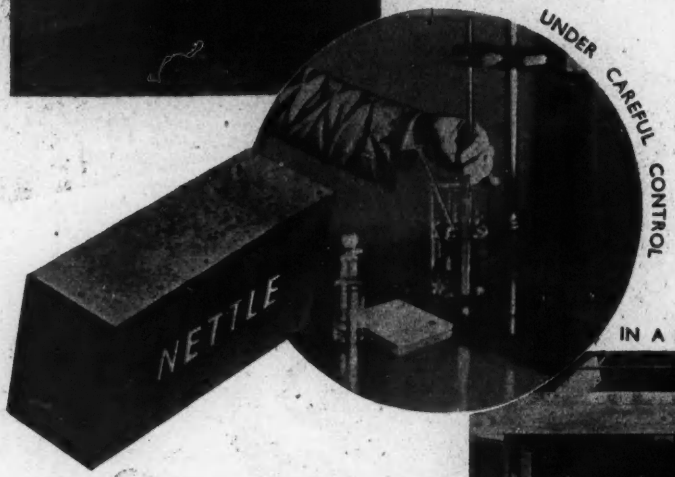

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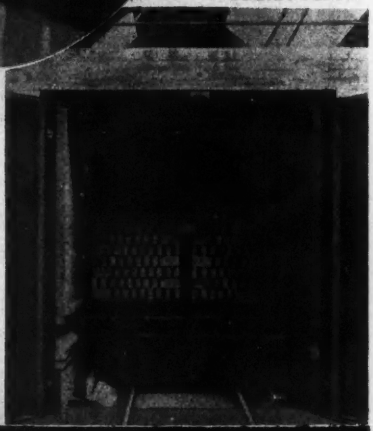

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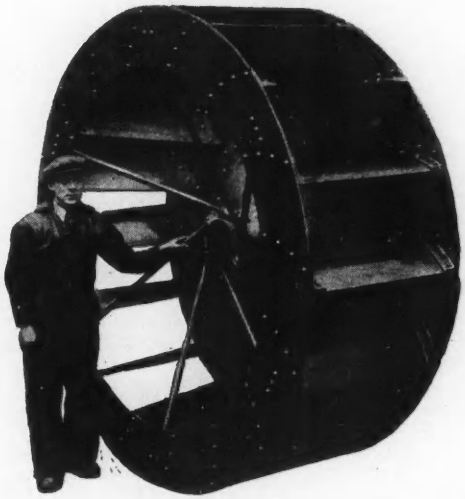

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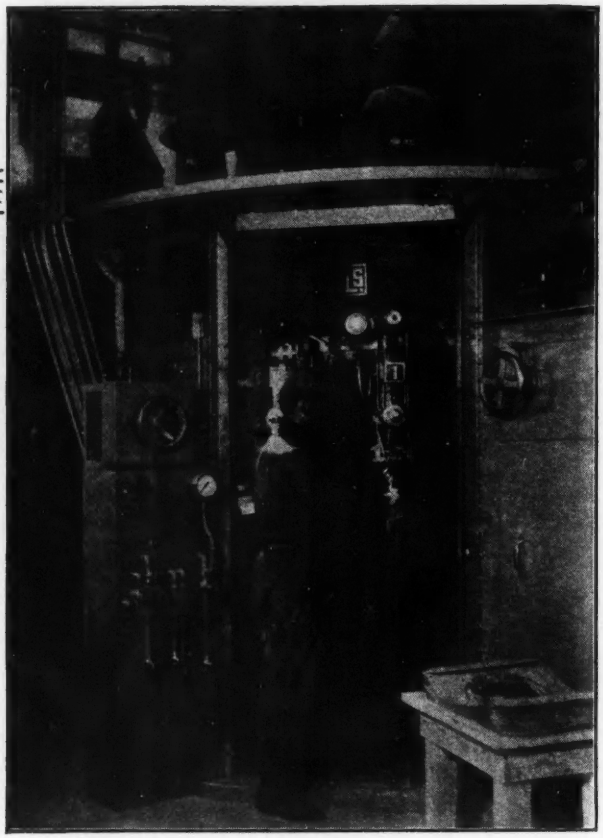

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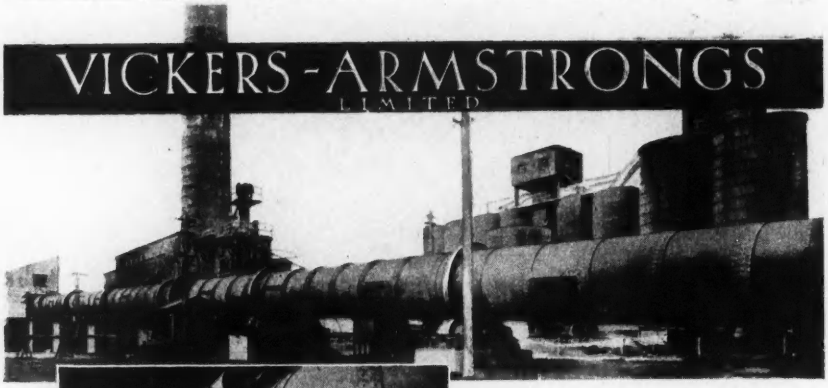

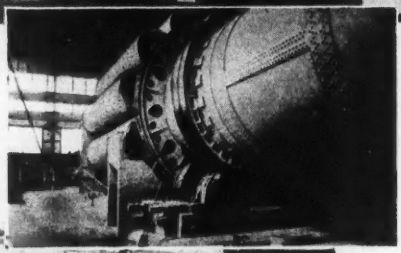

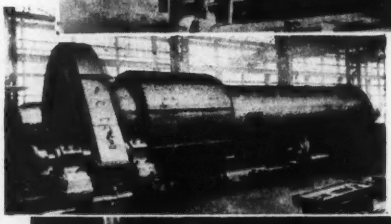

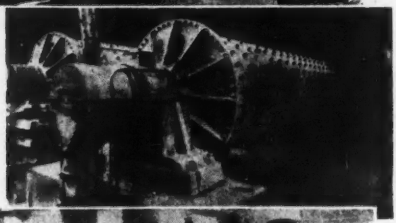

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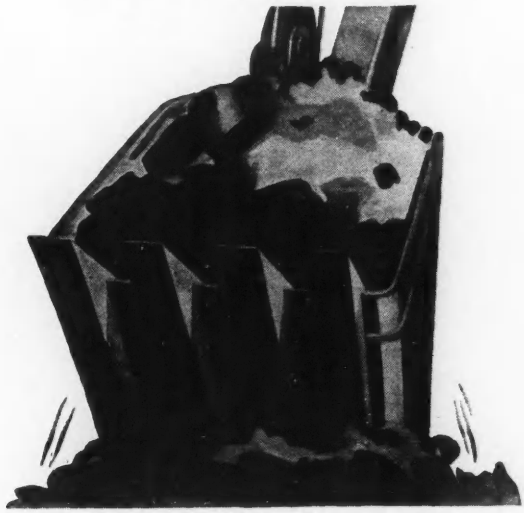

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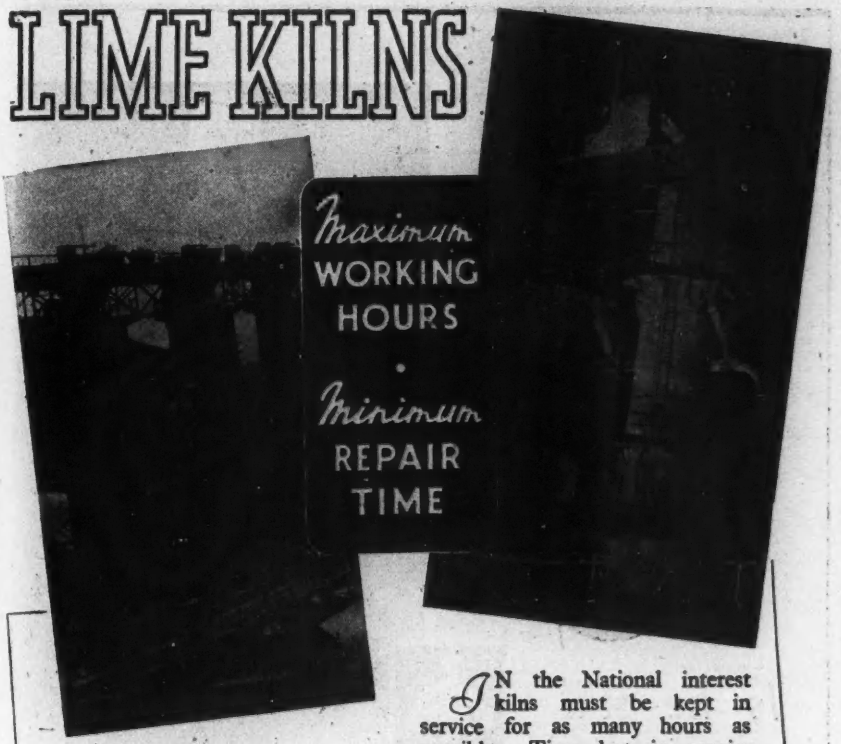

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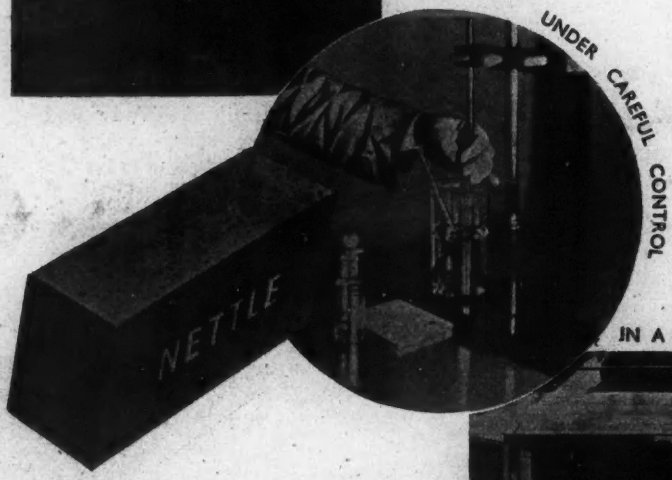

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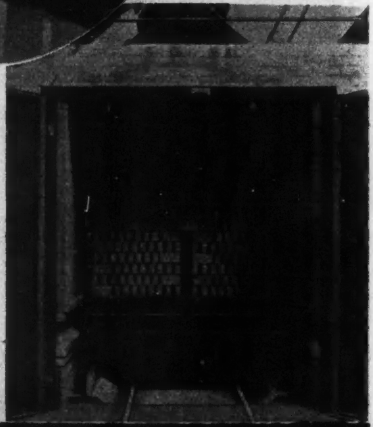

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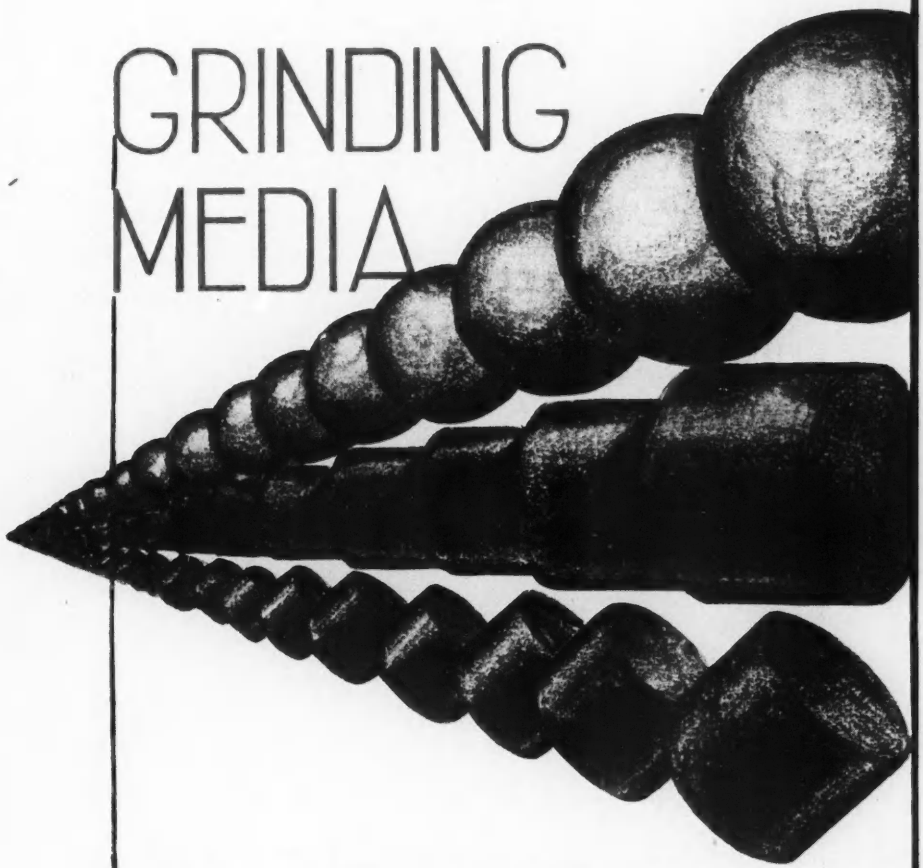

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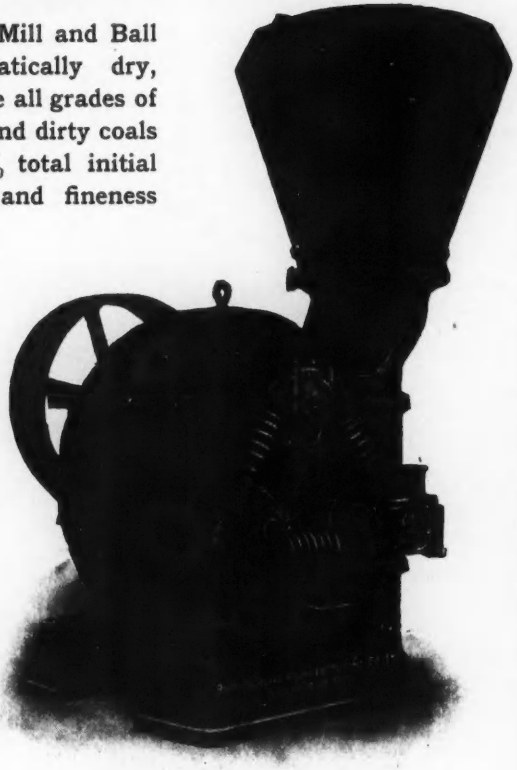

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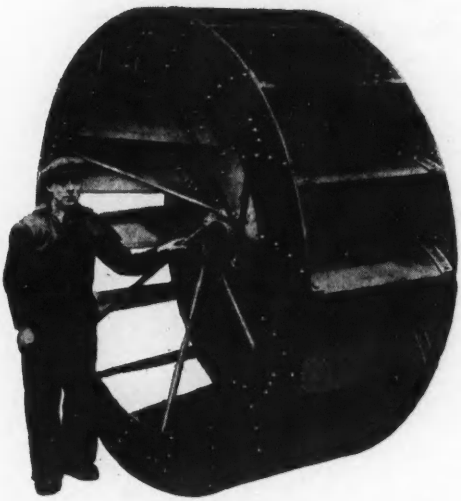

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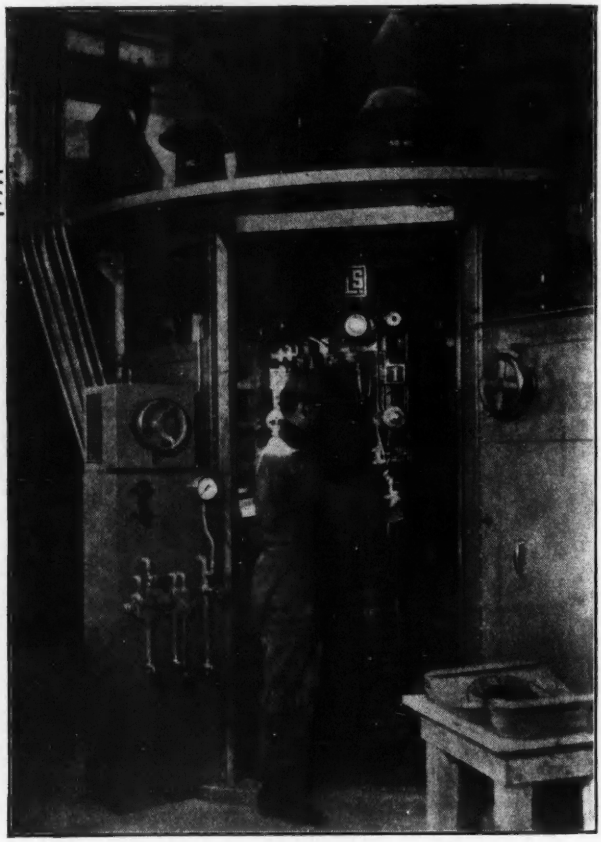

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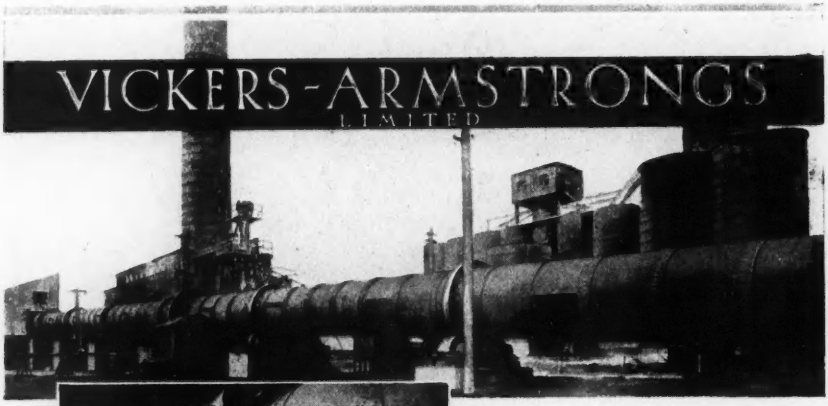

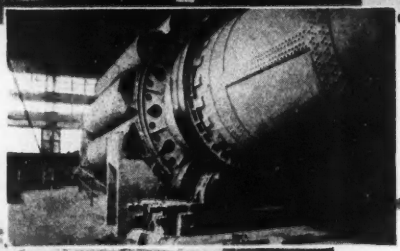

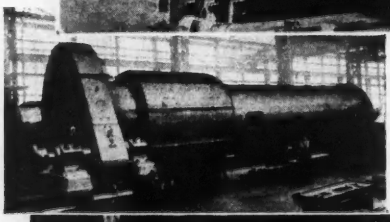

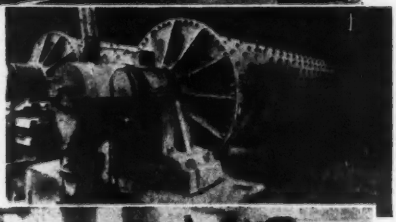

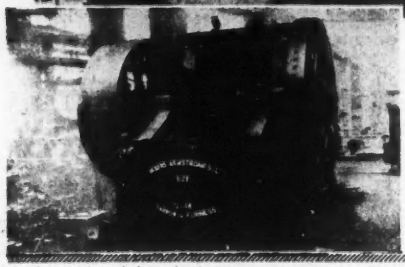

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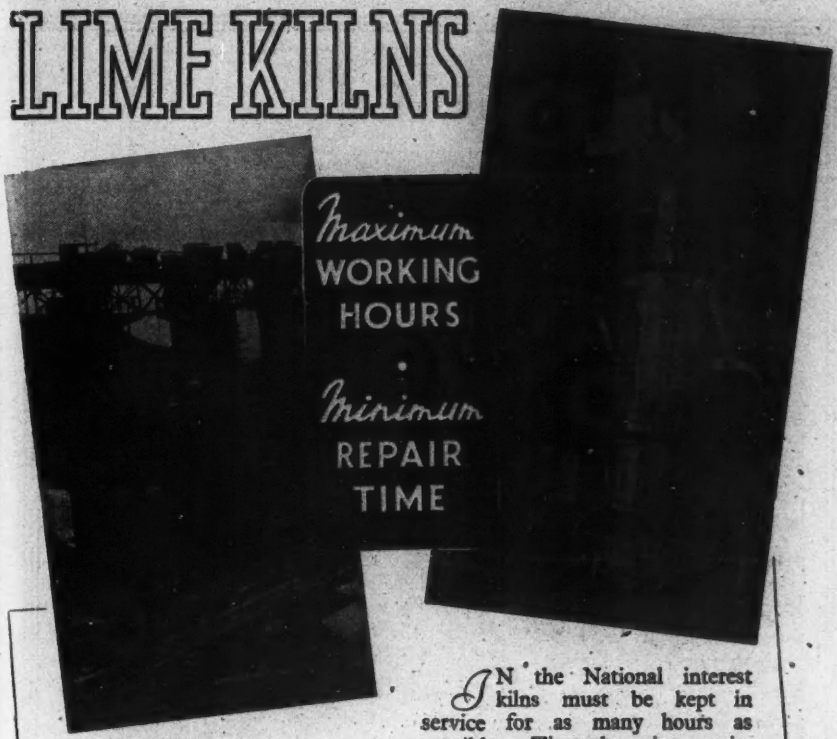

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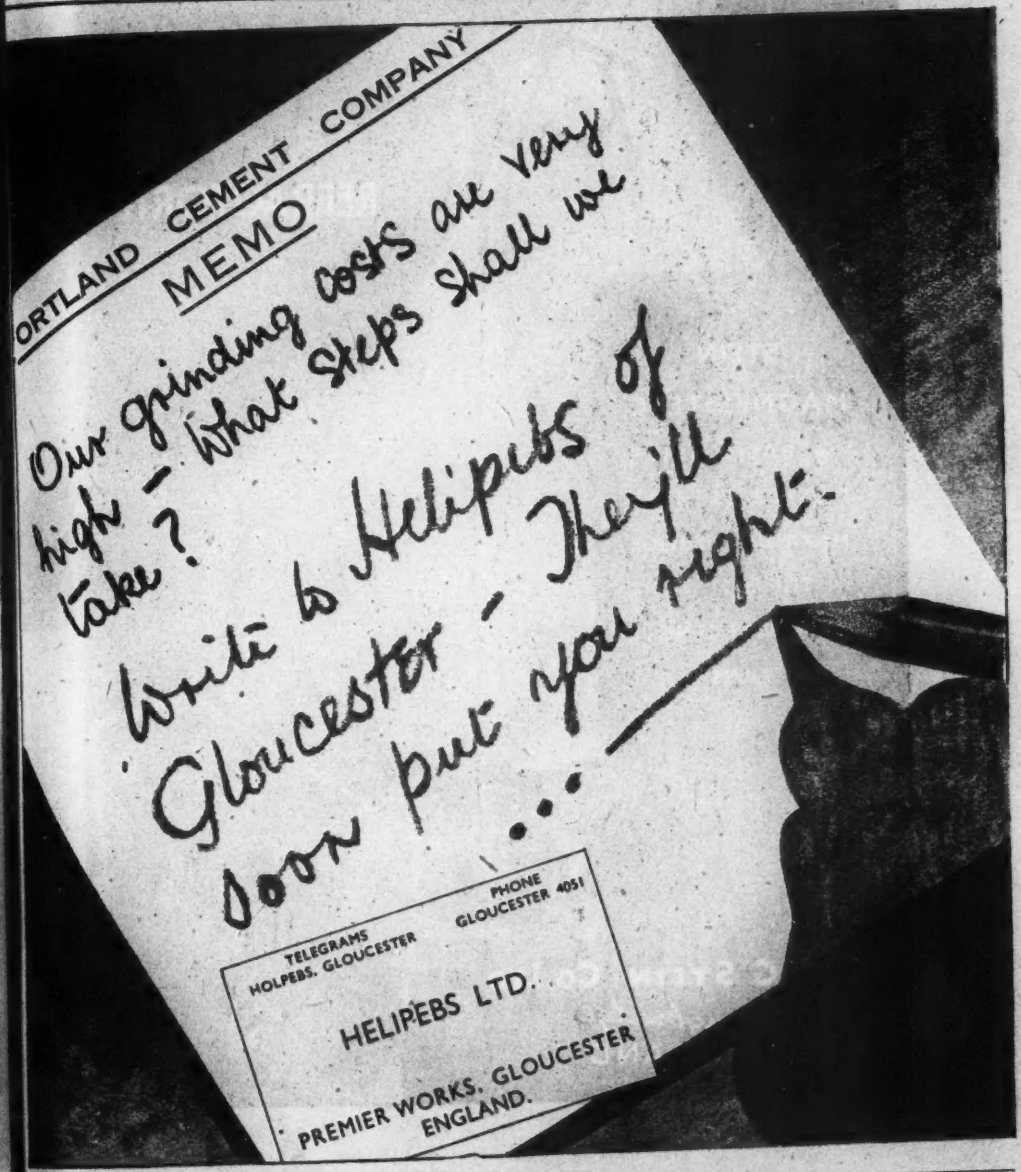
CEMENT AND LIME MANUFACTURE

Vol. XIX. No. 6 NOVEMBER 1946 Price 1/3 Alternate Months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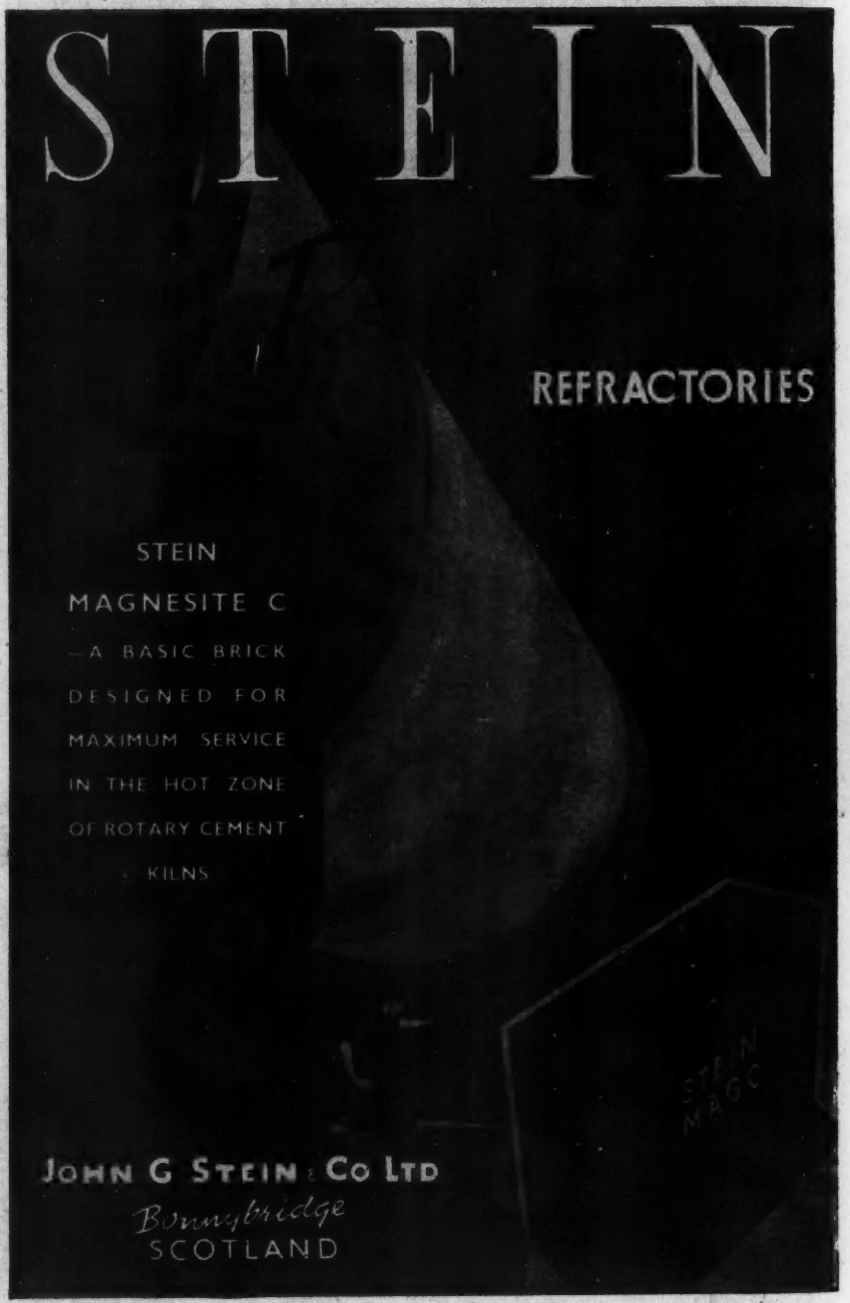
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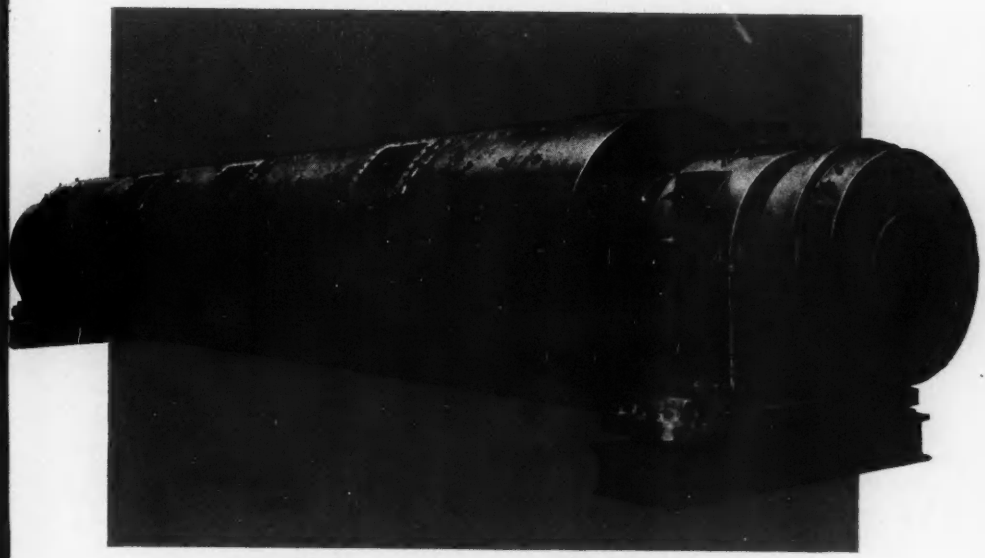

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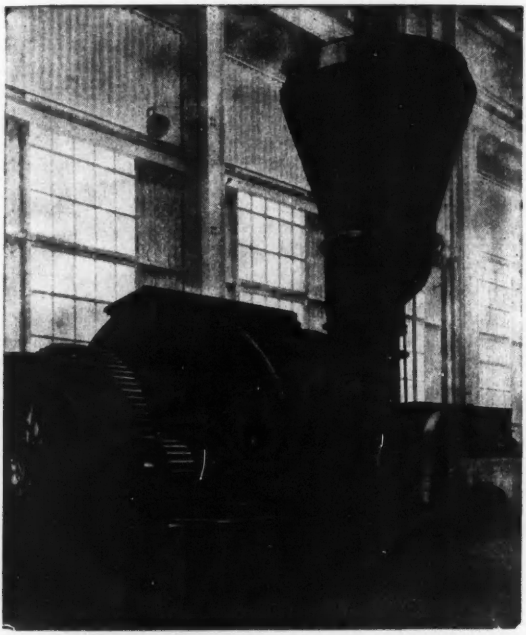

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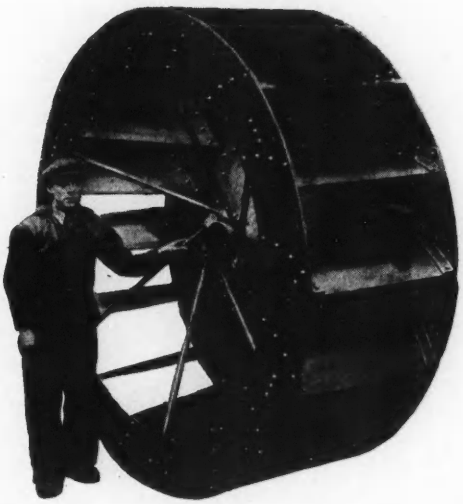

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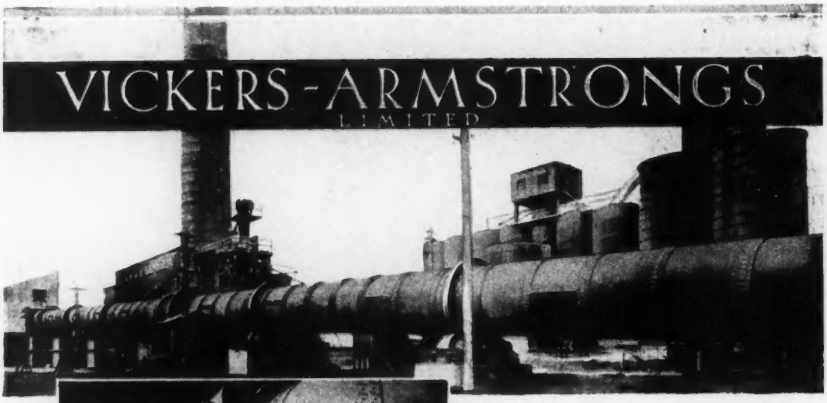

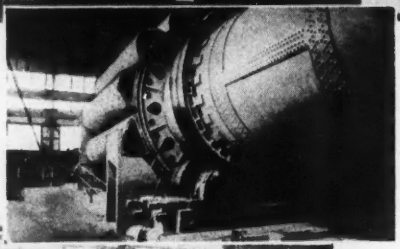

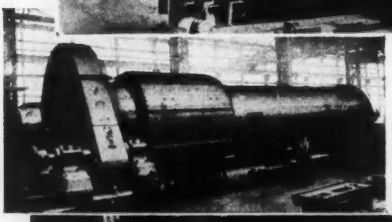

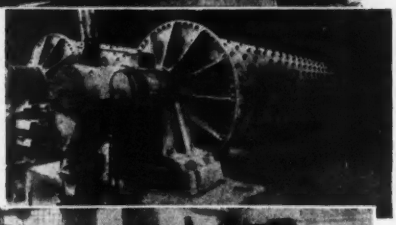

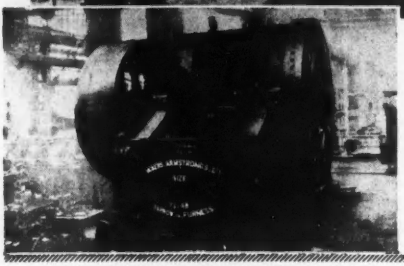

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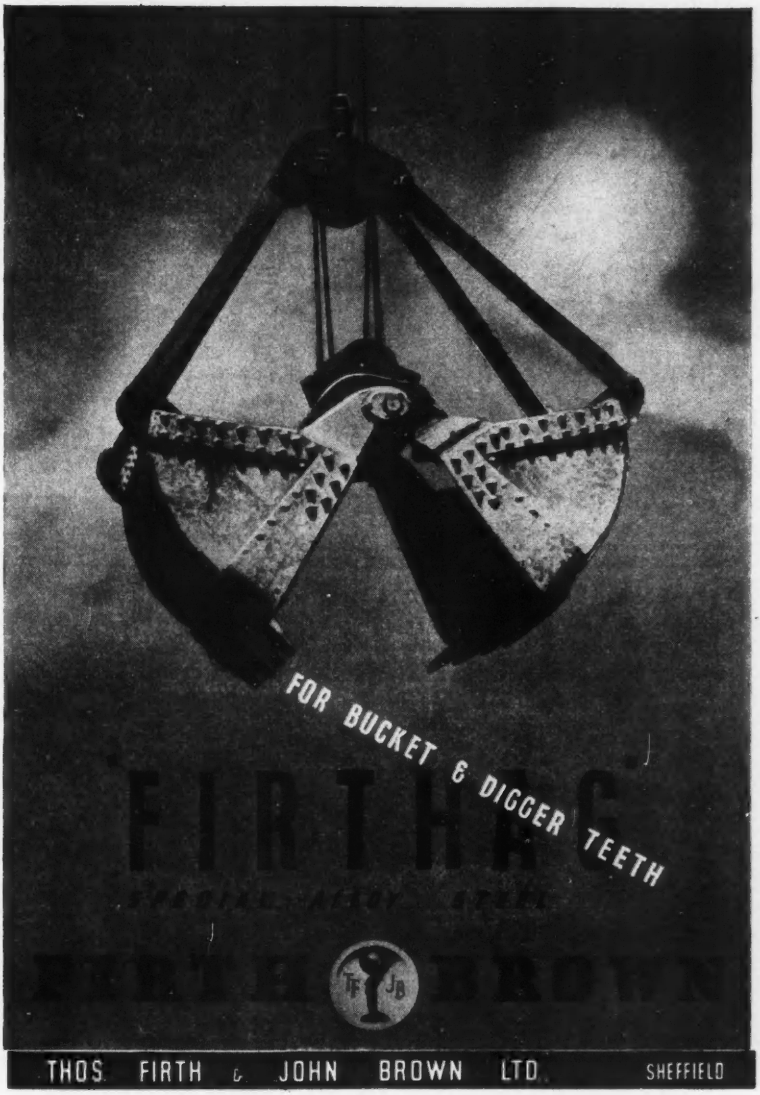
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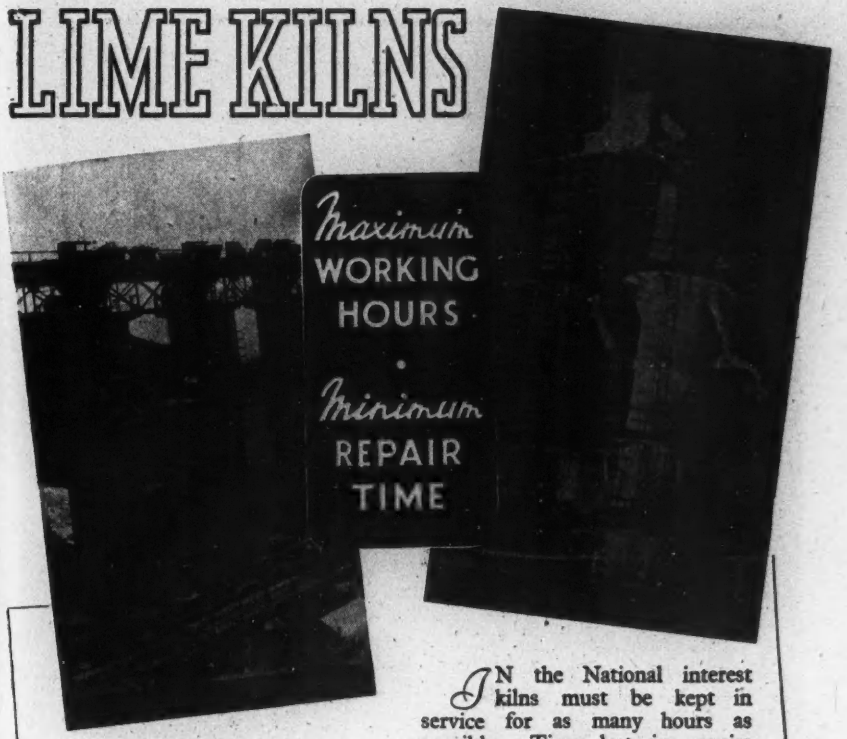

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